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Journal

—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries

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CONTENTS

	Page
Editorials:	529
The New Secretariat	531
Plans of the New Secretary	
Original Papers and Discussions:	536
The Composition of Barium Glass—Montgomery	
The Metallography of the Oxy-Acetylene Weld as Affected by Enameling—Poste	546
The Separation of Lime from Dolomite—Schurecht	558
Plastic Magnesia Cements—Bates and Young	570
Design of a Furnace for Annealing Optical Glass—Sharp	597
The Transverse Strength of Fire-clay Tiles at Furnace Temperatures	608
Activities of the Society:	616
New Members Received during May, 1921	

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NEW YORK

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JOURNAL

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AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Editor: E. W. WASHBURN; **Associate Editors:** L. E. BARRINGER, A. V. BLEININGER, R. L. CLARE, E. P. POSTE, H. RIES, F. H. RHEAD, R. B. SOSMAN, R. T. STULL, E. W. TILLOTSON

Vol. 4 **July, 1921** **No. 7**

EDITORIAL

THE NEW SECRETARIAT

The long contemplated event has been consummated. The Society through its Board of Trustees has taken the step that should assure continued development along the line of larger service.

Under contract dated July 1 and approved by the Board of Trustees July 8, Mr. Ross C. Purdy was employed as Organizing Secretary for the remainder of the year. At the beginning of the ensuing year he will become the General Secretary of the Society in which office the duties covered by the term Organizing Secretary will also be absorbed.

This decision was reached only after careful consideration of expressions from the Society including a favorable report by the Committee appointed at the last annual meeting and communications from a large number of the Society's membership.

It is well known that many in close contact with the Society's problems have been looking forward to the time when the employment of a full-time Secretary could be undertaken. They have regarded the creation of this office as an event in the normal program of development. This move is not one in which spectacular membership campaigning, or mushroom activities

will be a feature. The office has been created largely to meet a need that is already being made manifest by requests coming from organizations of industries themselves, indicating a service in which the Society must at once take leadership or jeopardize rightful position in its relation to the clay industries. I refer to the field that may be included under the term Coöperative Research. From this field the Divisions that flourish must draw their sustenance. Perhaps in some instances this must also provide new life to Local Sections.

The duties of the Secretary are not confined to the activity above emphasized, his duties being those prescribed by the special committee, later including those of general secretary, also "Such other duties as may be from time to time prescribed."

In the creation of this office the Society should be able to properly organize and supervise its underlying and fundamental activities. This, done in proper relationship with the industries, the problems of membership, finance, contributions to the Journal, and general interest and support, will find ready solution.

In forwarding this most important service the Society is very fortunate in securing the services of Mr. Purdy, who in the words of the special committee, "possesses the necessary qualifications of ability, leadership, zeal and energy to the fullest extent."

However, let no one of our members and officers presume that the burden of responsibility to the Society is lessened. The same necessity for personal interest, and devotion of thought and effort exists as before. The Society now has employed under the direction of the Board of Trustees an agency whereby the power to do and to serve is markedly increased.

The earnest coöperation of all concerned will enable the Society to rise to new and higher standards of attainment.

F. K. PENCE

PLANS OF THE NEW SECRETARY

In response to the requirements of the times and the expressed desire of the Society, the Board of Trustees has employed a Secretary to serve the remainder of this calendar year as Organizing, and next year as General Secretary. The object of employing an Organizing Secretary is none other, neither more nor less, than the object for which the Society was founded, and for which, for twenty-two years, it has been maintained:—"to advance the ceramic arts and sciences."

The organizing work of this secretary, as distinct from that which Prof. Binns is so efficiently doing, can be generalized in the words of the special committee in its report to the Board of Trustees, which report was accepted, and approved, and the section under caption—"Duties of the proposed permanent Secretary"—made a part of the contract of employment.

These, in part, are:

It should be the duty of the Organizing Secretary to assist the officers of the divisions in planning the work and executing the tasks undertaken. He should report to the Board of Trustees upon the status of the divisions at the expiration of every quarter. He should be charged with the duty of supporting and supplementing the work of the Committee on Membership and may be authorized by the Board of Trustees to undertake and prosecute such additional measures which promise to increase the membership of the Society, both individual and corporate.

It should be the duty of the Organizing Secretary to assist the Committee on Papers and Program in securing contributions of merit and in every way possible, to aid in improving the program of the meetings and bringing before the membership topics of vital and timely interest.

It should be the duty of the Organizing Secretary to work towards the establishment of coöperative researches, conducted under the auspices of the divisions of the Society, and supported by associations or groups of manufacturers, or single corporations. He should be charged with the duty of preparing research agreements, subject to the approval of the Board of Trustees. All disbursements made from funds made available to the Society for research purposes should be made by the Treasurer.

It should be the duty of the Organizing Secretary to aid the Division Officers in formulating research problems. It should be his duty likewise to supervise the prosecution, reporting, and publication of such researches, unless other supervision is provided for by the Division of the Society, and to report to the Board of Trustees, upon the progress of such work monthly.

It should be the the duty of the Organizing Secretary to maintain friendly relations with the associations of manufacturers and similar organizations, to address them on the subject of the work of the Society and related topics and to secure their coöperation in our several fields of endeavor.

This officer should be charged also with the duty of assisting in the preparation for meetings of the Society, the securing of suitable quarters and necessary equipment.

It is important to note that the scope of the Organizing Secretary's authority in all these matters is limited to "assisting," "supplementing," "supporting" and "aiding." He is to encourage, promote and execute only by the authority of, in the name of, and through the Board, the Committees, the Local Sections or the Divisions, as each case will require. In other words, he is merely to assist, support, aid and supplement the existing agencies already provided for by the Constitution and By-Laws.

The Organizing Secretary is a representative of the Board of Trustees, and therefore, such authority as may be possessed by him shall be only that granted to him by the Board. This same limitation is placed on all the executive officers of the Society: the President having "general supervision of the affairs of the Society under the direction of the Board of Trustees,"—and—"can countersign checks drawn by the Treasurer when such drafts are known by him to be proper and duly authorized by the Board of Trustees."

The Standing Committees, Local Sections and Divisions have duties and authorities given by the Constitution and By-Laws that are their own, and of kinds that are wholly apart and distinct from those possessed by the Board. But, the Constitution says that "on failure of any officer or any member of a committee to execute his duties within a reasonable time, the Board of Trustees, after duly warning such person, may declare the office vacant and appoint a new incumbent." With the possession of this authority on part of the Board there is a corresponding responsibility of seeing to it that the Committees, Local Sections and Divisions perform their tasks, hence it is well that the Board, through its representative, the Organizing Secretary, shall "assist," "advise" and "supplement" in whatever is undertaken by the Committees, Sections and Divisions.

By the appointment of an Organizing Secretary to serve in the capacity of an assisting and supporting supervisor of the work of the Committees, Sections and Divisions, the Board of Trustees, for the first time, has a means of coördinating and unifying the work of the entire organization on the one general program of advancing the ceramic arts and sciences. That distribution of responsibilities, and those unrepressed and unstinted opportunities for service by each independent unit in the organization, which should and which do characterize this Society, is in no way curtailed. On the other hand, there is assured, as far as it is humanly possible so to do, to each Industrial Division, Section and Committee, that freedom of development along lines of its own choosing, which is so essential to holding the interest of, and obtaining the full support of, the individual members without danger of any of them losing sight of the common interests and mutual purposes for which they have joined together in support of the Society as a whole.

Emphasis should be placed on the fact that the Organizing Secretary can not and will not relieve the Committeemen or the Officers of the Local Sections, and Divisions, of their responsibilities. The limit of the Secretary's activities is assisting, supporting and promoting; in other words, a general supervision of the technical work of the Society.

The Board is more concerned with the general business of the Society, while the Committees, Sections and Divisions are engaged in specific fields more directly concerned with attaining the ultimate object of the Society, that of "advancement of the ceramic arts and sciences." This division of responsibilities gives the Society two groups of executive officers, (1) the general supervising and managing (the Board) and (2) the technical (Committees, Sections and Divisions).

The need is recognized for the full time service of an officer to assist the Committees, Sections and Divisions (not to do their work) in such service as will build the Society up to the strength and the position of being recognized by the ceramic industries and by the other technical associations, and the Bureaus and Colleges as the pivotal unit, the prime mover in the promo-

tion of ceramic arts and sciences as it was in the earlier years under less developed conditions. This necessitates the keeping of each and every member in close working contact with the Society, and the Society, in turn, in close working understanding with the other ceramic associations and the users of ceramic products, coöperation with other national technical organizations in those endeavors in which the ceramic groups have reason to be interested.

One such activity is coöperative research. Research financed by associated corporations is in vogue today, whereas in former years the industries supported general ceramic research without regard to special applications. As the industries progressed in things technical, they learned the value of joining by groups in financial support of researches in which they were particularly interested. Thus their support of and interest in scientific and technical research gradually shifted from general to specialized investigations. The advantages of group specialization, emphasized as they were by war conditions, was recognized by the American Ceramic Society through its establishment of Industrial Divisions.

These Divisions have succeeded or failed according to the quality and the continuity of executive attention given by the Division Officers, and also according to the extent to which they have enlisted their particular group of manufacturers in the support of scientific and technical research. It is to correct these shortcomings that the Organizing Secretary was appointed, for the success or failure of the Divisions is the success or failure of the Society as a whole in serving the different groups of ceramic industries.

The American Ceramic Society has been and will continue to be the principal agency for the advancement of ceramic arts and science. In the early days when there was but the one collegiate department of ceramics, no federal bureaus, and when the manufacturers were jealous of their trade secrets, Prof. Edward Orton, Jr. did a great service to the Ceramic Industries when he persuaded a few ceramic workers to join in the founding of the American Ceramic Society. Since 1899 this Society has continued steadfast to its original purpose, and was and is de-

serving, and enjoying the support of the Ceramic Industries with the result that today our Industries are second to none and equal to any in the world in technical attainments.

It is intended that this Society shall continue as the leading agent for the advancement of Ceramic Technology, and it is assured that each Division will enjoy in this purpose the full coöperation and backing of its respective group of manufacturers with the same zeal and for the same purposes with which, in previous years, the Society as a whole was supported.

That all this may be realized and thus the purposes and hopes of Prof. Orton and his collaborators carried to full fruition, the Board of Trustees has elected a full time Secretary and given emphasis to the organizing duties as distinct from the routine; and it is to the carrying through of this program that I, the elected Secretary, pledge my ability, zeal and singleness of purpose, and it is to this that the full coöperation of each and every member of the Society is asked.

ROSS C. PURDY

ORIGINAL PAPERS AND DISCUSSIONS

THE COMPOSITION OF BARIUM GLASS[1]

By Robert J. Montgomery

ABSTRACT

In this paper the relation between the composition of barium glasses and the optical constants is discussed as well as the general rules for proportioning batches. The important results and conclusions are given by the figures. After the required amounts of BaO and B_2O_3 have been obtained from figures 2 and 3, the amount of SiO_2 can be obtained from figure 5, the alkali and ZnO from figure 7 and the Al_2O_3 from figure 8. If the total exceeds 100, the ZnO and the Al_2O_3 may be reduced. The proportions cannot be as definitely expressed as in the case of lead glasses and more experience is required to enable one to change the optical properties at will.

Introduction

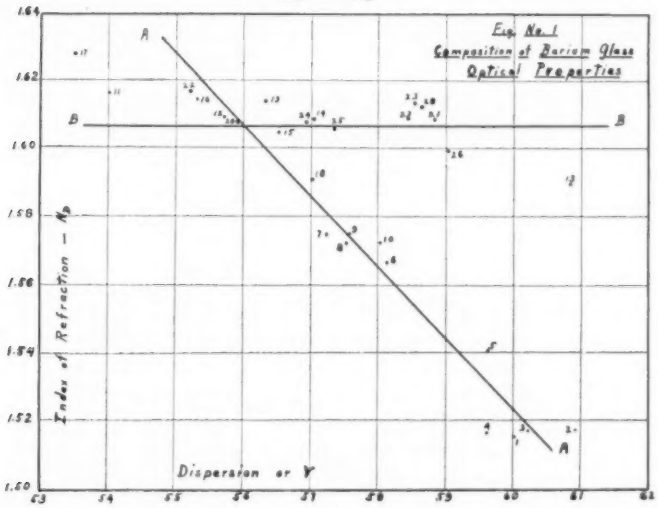
In a previous paper,[2] the optical glasses whose main component is lead were discussed. The present paper covers another section of the optical glass field and includes those glasses whose properties are dominated by their barium oxide content. The composition of barium glasses is not as easily covered as that of lead glasses, because of the greater complexity of the batch. With lead glass we have essentially a three component system of alkali, lead and silica, while in barium glasses we have a second acid (B_2O_3) introduced, as well as Al_2O_3 and a more comlpicated RO containing zinc and calcium in addition to alkali and barium. The zone covering the relation of optical properties to composition, which is narrow in the case of the lead glasses, is here much wider with a dispersion, as expressed by the ν value, ranging from 54 to 61 and an index of refraction for the D line, from 1.50 to 1.63. The composition is further complicated by the use of lead in the baryta flint

[1] Received April 2, 1921.

[2] This Journal, 3, 900 (1910)

glasses. These lie between the typical barium glasses and the lead glasses proper and their composition will not be discussed in this paper. In the paper on lead glasses referred to above the compositions were discussed on the equivalent weight basis because of their simplicity. With barium glasses it was found that little or nothing was gained by employing the equivalent weight basis because of the complexity of the glasses. There are no simple relations between composition and optical properties and the relations observed have to be expressed in more general terms. For this reason the composition will be given in weight per cents

$$\nu = \frac{N_D - 1}{N_F - N_C}$$



of the oxides present. The optical properties referred to are the refractive index for the D line, N_D, and the reciprocal dispersion or constringence ν, defined by the equation.

In figure 1 are plotted the optical properties of 28 typical barium glasses whose compositions are given in table 1. These extend from the ordinary crown glass as a base through the barium silicate crowns, and the dense barium crowns to the densest barium crown. The relation of these glasses to the general field may be seen by referring to the chart given in a previous paper.[1] The baryta

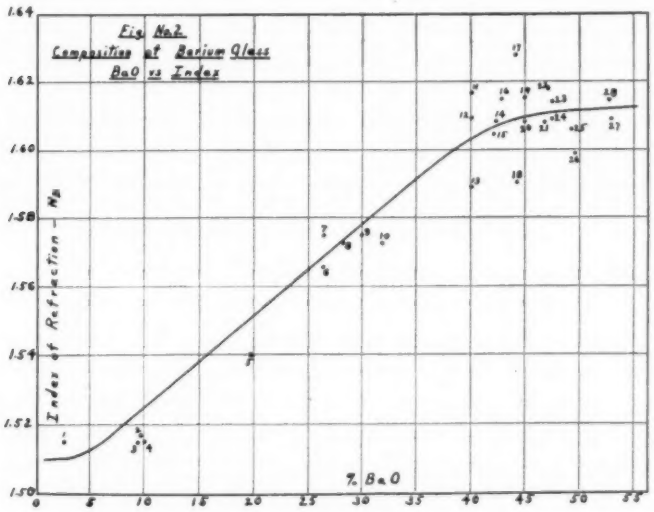
[1] THIS JOURNAL, 3, 404–10(1920).

TABLE I

No.	SiO_2	B_2O_3	K_2O	NaO	BaO	CaO	ZnO	As_2O_3	Al_2O_3	PbO	N_D	ρ
1	71.56	...	9.01	8.08	2.61	8.02	...	0.72	...	...	1.5160	60.0
2	68.50	3.50	5.00	12.00	9.70	...	1.00	0.20	...	...	1.5179	60.9
3	64.60	2.70	15.00	5.00	10.20	...	2.00	0.40	...	...	1.5179	60.2
4	67.00	3.50	5.00	12.00	10.60	...	1.50	0.40	...	...	1.5171	59.6
5	59.13	3.04	9.70	3.16	19.25	0.13	5.00	0.34	0.11	...	1.5409	59.6
6	49.57	4.51	8.49	1.01	26.06	...	10.03	0.34	...	...	1.5666	58.1
7	48.10	4.60	8.00	2.00	26.05	2.00	8.40	0.50	...	...	1.5755	57.2
8	48.10	4.50	7.50	1.00	28.30	...	10.10	0.40	...	...	1.5726	57.5
9	48.00	4.50	6.10	2.00	29.50	...	10.00	1.40	...	...	1.5754	57.5
10	48.45	3.65	7.55	0.95	32.63	...	6.46	0.32	...	...	1.5727	58.0
11	37.23	6.02	...	3.19	40.40	...	10.79	0.37	1.98	...	1.6167	54.0
12	37.30	6.00	...	3.20	40.50	...	10.80	...	2.00	...	1.6097	55.7
13	37.50	15.00	...	...	41.00	...	...	...	5.00	...	1.5899	60.8
14	34.50	10.10	...	...	42.00	...	7.80	0.50	5.00	...	1.6088	57.0
15	39.63	6.04	...	...	42.08	...	9.24	0.50	2.50	...	1.6054	56.5
16	36.28	5.57	3.20	...	43.62	0.21	10.73	0.39	...	...	1.6147	55.3
17	37.00	5.00	2.60	...	44.00	...	7.70	0.70	...	3.00	1.6242	53.5
18	39.60	5.00	...	...	44.00	2.00	7.70	0.40	...	3.00	1.5905	57.0
19	39.00	5.00	...	...	45.00	2.60	7.00	0.40	1.00	...	1.6145	56.3
20	37.80	5.69	3.50	...	45.04	...	7.56	0.40	...	...	1.6085	55.9
21	34.56	10.96	0.09	0.21	46.91	...	1.14	0.59	5.02	...	1.6091	58.8
22	37.00	5.00	2.60	...	47.00	...	7.70	0.70	...	...	1.6177	55.2
23	32.27	12.90	0.33	...	47.20	...	1.12	0.20	6.10	...	1.6139	58.5
24	38.29	5.76	2.84	...	47.60	...	5.11	0.41	...	...	1.6085	56.9
25	38.12	7.18	2.40	...	49.37	...	2.54	0.41	...	...	1.6062	57.3
26	36.58	11.60	0.10	0.22	49.66	...	1.21	0.62	...	...	1.5991	59.0
27	36.46	8.91	1.21	...	52.88	...	...	0.39	...	...	1.6091	58.4
28	36.51	8.93	1.22	...	52.96	...	...	0.39	...	...	1.6138	58.6

References—C. N. Fenner, THIS JOURNAL, **2**, 102(1919).
Williams and Rand, *Ibid*, **2**, 422(1919).
Allen and Zies, *Ibid*, **1**, 739(1918); Doelter's "Handbuch der Mineralchemie."

light flint, telescope flint and soft silicate crown are omitted because of the lead content although they contain considerable barium. Only one barium silicate crown glass was available but the others used are selected from a great many obtained from experimental melts in our plant, and from published information. On first consideration the scattered positions of the points seem to show little regularity. A further study reveals certain underlying relations and these will now be discussed.



BaO vs. Optical Properties

When it is desired to increase the index of refraction in a soda-lime glass without lowering the ν value, barium is used. The dispersion is not affected to any extent by the barium and we may have more than 40 per cent BaO in glasses whose ν values vary only from 53 to 60. The line A-A in figure 1 shows the effect of the addition of barium to a soda-lime glass. The ν value decreases not because of the increased barium but because of the decrease in SiO_2 which the barium displaces. The normal index of refraction of a soda-lime glass is about 1.51, largely due to its content of silica which has an index of 1.4585. It may be raised as high as 1.52 to 1.53 by the addition of lime, but above this point we have to

Allen and Zies, *Ibid*, 1, 739(1918); Doelter's "Handbuch der Mineralchemie."

resort to barium if the ν value is not to be lowered. The relation between the increase of index and the barium content is very nearly a straight line as the barium increases up to 40 or 42 per cent. This relation is shown graphically in figure 2. Above 42 and up to 54 per cent, which is about the maximum in these glasses, very little change in index is noted, the line becoming practically horizontal. The points do not lie as closely to the line as could be wished, but as the index is affected by the other constituents the variation is not unexpected. Glass number 17 contains 3 per cent of PbO which is responsible for its rather high index. Barium cannot be studied without the introduction of B_2O_3, since with the higher per cents of BaO, boric acid is necessary in order to prevent crystallization on cooling. The effect of B_2O_3 will therefore be shown next in conjunction with SiO_2 as both have to be considered together in a discussion of optical properties.

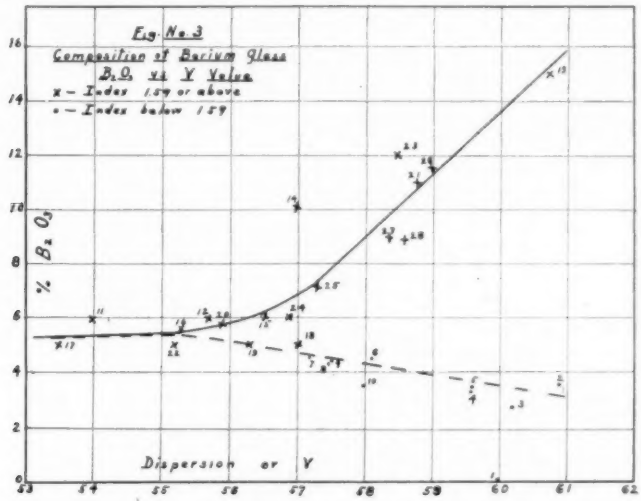
B_2O_3 and SiO_2 vs. Optical Properties

In considering the effect of these two materials their optical properties must be kept in mind. They are as follows: Boric acid glass N_D 1.463, ν 59.4; Silica glass N_D 1.4585, ν 67.9.

Their influence on the optical properties of glass are quite consistent except in one case. In a high BaO glass, SiO_2 will lower the index more than will B_2O_3. For some unknown reason an addition of B_2O_3 will often *raise* the index of refraction in both barium and lead glasses when the index is about 1.60. It very seldom causes a drop in index although the index of boric acid glass is low.

Soda-lime glasses and glasses of this general type containing small amounts of barium are dominated by the properties of silica, and have an index of about 1.518 and a ν value of about 58 to 60. The effect on the dispersion, of the B_2O_3 which may be present up to 5 per cent, is not noticeable. In the glasses high in BaO, which have a correspondingly low SiO_2 content, the effect of B_2O_3 is more easily seen. It is quite apparent when the index is 1.59 or above. The line B-B in figure 1 shows the tendency of B_2O_3 to increase the ν value when the SiO_2 content is constant. In figure 3 the relation between B_2O_3 and the dispersion is given. Glasses with an index of 1.59 or above are indicated by crosses and the heavy line curve passes through these points. The

glasses containing less than 6 per cent B_2O_3 evidently fall into another group indicating that the higher ν value may be obtained with silica as well as with B_2O_3. A dash line passes through this group. The variation in dispersion is evidently limited by the optical properties of SiO_2 and B_2O_3 and the amount of each which can be introduced into a commercial glass.



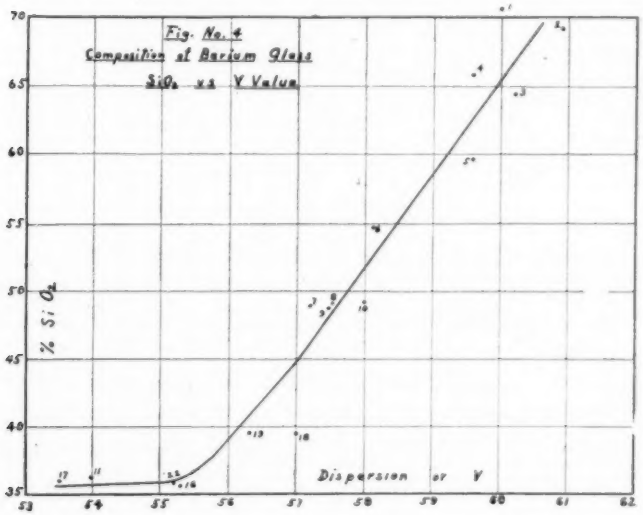
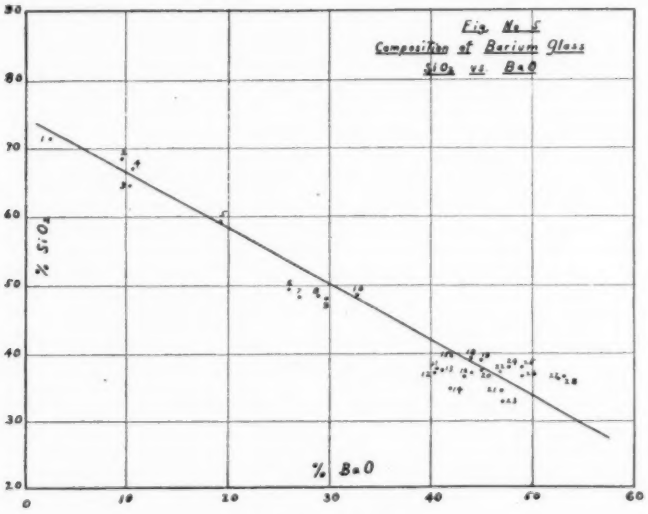


Figure 4 shows the relation between SiO_2 and the ν value in those glasses containing less than 6 per cent B_2O_3. This includes the group through which the dash line is drawn in figure 3.

There are a number of general relations referring to the proportioning of the constituents of the batches which may be shown in curve form. These are as follows:

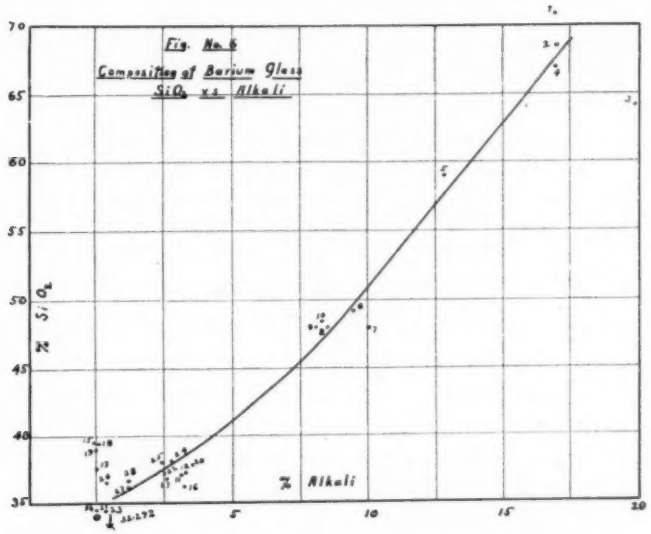
SiO_2 vs. BaO

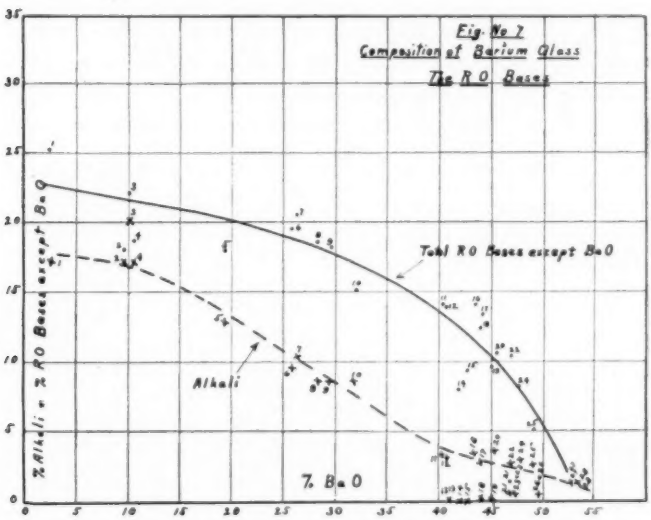
The relation between the barium and silica content is clearly shown in figure 5. The required proportions are quite well defined, a decided drop in the silica content as the barium increases being noticeable.



SiO_2 vs. Alkali

As would be supposed, the amount of alkali in the glass increases with the silica. The curve in figure 6 shows this relation. While the points are not all very close to the curve drawn the general relation is beyond question. It must be remembered that in these complex barium glasses a comparison of any two constituents will be affected, often to a considerable extent, by the other constituents present. Only general relations can be established.





The Proportioning of the RO Bases

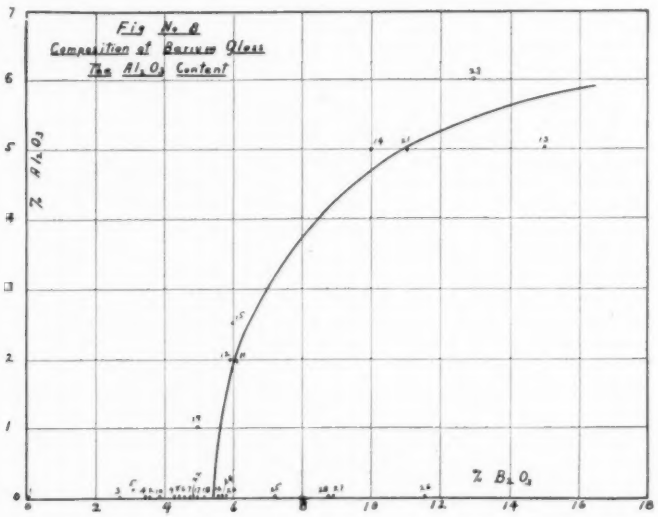
In figure 7 the relation between BaO and the other RO bases is shown. The solid line goes through the total RO bases except BaO and includes K_2O, Na_2O, CaO, ZnO and PbO. The dash

line runs through the points representing the alkali content including K_2O and Na_2O. The positions of the two curves are fairly well defined except when the BaO is over 40 per cent. Some of the the glasses contain no alkali while others contain 2 to 3 per cent. Alkali is not necessary to obtain the desired optical properties and when used it is for the purpose of controlling the melting and working properties of the glass.

The Al_2O_3 Content

It is hard to determine just what the Al_2O_3 content depends on. It is not necessary to use alumina to control the optical properties but it is almost invariably used and the amount seems to depend upon the B_2O_3 content, at least an interesting comparison can be obtained between the amounts of these two constituents. Alumina would tend to increase the viscosity of the glass and reduce the attack upon the pot.

In figure 8 the Al_2O_3 and B_2O_3 contents are plotted against each other and an average curve is given. Al_2O_3 is seldom used until the glass contains at least 5 per cent B_2O_3 and then it increases quite rapidly with the addition of B_2O_3 reaching a maximum at about 6 per



cent. Points 25, 26, 27, and 28 are not on the curve and they are rather abnormal in this respect. They prove that the optical properties do not depend upon the Al_2O_3.

The ZnO Content

Zinc oxide may run as high as 11.0 per cent in these glasses but it has little effect upon the optical properties. It is used largely to fill in when it is undesirable to increase any of the other constituents. Zinc oxide may replace BaO to lower the index of refraction or replace B_2O_3 to lower the ν value.

Alkali

Potassium is the alkali almost invariably used because the sodium compounds give greater color. The amount used depends upon the flux required after the SiO_2, BaO, B_2O_3 contents are adjusted to give the proper optical constants.

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THE METALLOGRAPHY OF THE OXY-ACETYLENE WELD AS AFFECTED BY ENAMELING[1]

E. P. Poste

ABSTRACT

Metallography of Low Carbon Steel.—A discussion of the cooling of steel through the critical range is followed by a consideration of the general structure of low carbon material. A brief discussion of mechanical and thermal treatments follows including the statement of the essential conditions for annealing.

Investigation of Oxy-Acetylene Welds.—The temperature reached by the steel in the enameling operation is discussed with reference to the annealing temperature. An examination of plates and welds before and after enameling shows that a thorough annealing of the weld has been accomplished.

Although the subject of metallography is undoubtedly new to the average enamel technologist, it will only be possible to very briefly cover some of the fundamentals before taking up the experimental work being covered by this paper. If the reader is interested in a more complete consideration of the principles of metallography, he will be able to make use of the following references:

The Metallography and Heat Treatment of Iron and Steel—Sauveur.
Metallography of Steel and Cast Iron—Howe.
Chemistry of Materials—Leighou. Pages 102–118.

Metallography of Low Carbon Steel

Steel is essentially an alloy of iron and carbon produced by certain definite metallurgical processes. In the melted condition, it consists of molten iron holding the carbon in solution. After solidification the mass assumes certain crystalline forms which undergo definite changes in cooling through what is termed the critical range, from approximately 900 to 770°C (1650 to 1420°F). After cooling below this range the steel is found to be made up of two substances termed "ferrite" and "pearlite;" the former is iron, while the latter is a definite combination of iron and carbon.

Confining our discussion to low carbon steels such as are used in enameling, it should be noted that the amount of ferrite greatly

[1] Received March 1, 1921.

predominates over the pearlite and a carefully polished and etched specimen observed under the microscope will show in general a net-work of more or less regular polygons of ferrite between which are areas of pearlite.

The structure just described is the normal structure existing after slow cooling. If the metal is cooled more quickly through the critical range or is worked upon mechanically at or below the critical range, the resulting structure may be materially different. Further, if metal originally below the critical range is heated to or above its critical range and recooled or worked mechanically and recooled, a great variety of results is possible. This general principle is the basis of all mechanical and thermal treatment of steel including hardening, tempering, annealing and similar operations.

The critical temperature of steel varies with the carbon content, the greater the percent of carbon the lower the temperature. It follows from this, therefore, that the temperature to which the piece must be raised to effect various types of heat treatment, such as annealing, must be in general slightly above the critical temperature for steel of the particular carbon content involved.

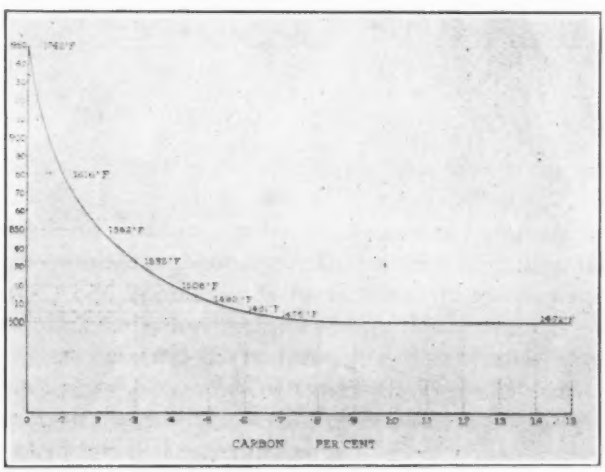

FIG. 1.—Annealing temperatures of steel. See "Metallography and Heat Treatment of Iron and Steel"—Sauveur. Page 234.

Figure 1 shows the minimum annealing temperature for the various carbon steels. With particular reference to the steel used in enameling, which generally falls between 0.10 and 0.20 carbon, it will be noted that the minimum annealing temperatures range from 880 to 850°C (1616 to 1562°F).

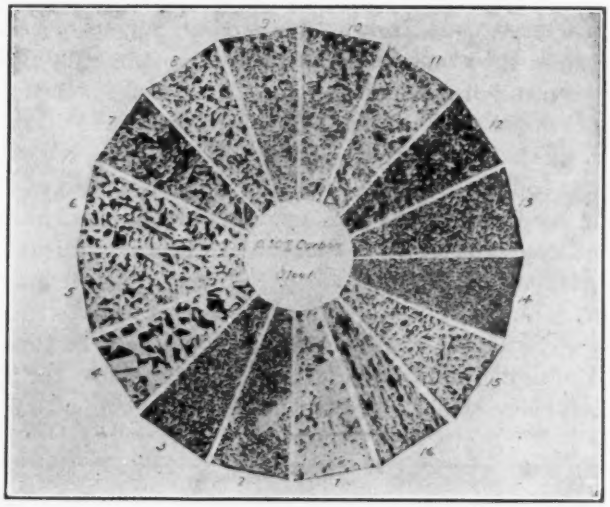

FIG. 2.—Various structures of 0.30% carbon steel. See "Metallography and Heat Treatment of Iron and Steel"—Sauveur. Page 253.

Figure 2 shows various possible variations in the structure of 0.30% carbon steel. Number 4 represents a very large grained structure produced by certain definite mechanical and thermal treatment while number 14 represents the other extreme of structure produced by another set of mechanical and thermal conditions. Numbers 1, 10 and 11 illustrate some indefinite structures similar to those which will be observed later in connection with the experimental work. Number 16 shows cold worked metal with "banded" structure similar to that which will be mentioned later.

In general a fine grained even structure is more ductile than a course or uneven structure; for instance number 14 in figure 2 is the desirable structure in a $^1/_2$ inch round rolled bar while numbers 5, 8, 9, 10, 11 and 12 are less desirable structures in the same bar.

Investigation of Oxy-Acetylene Welds

Various observations in connection with the firing of enameled tanks have long indicated that the enameling operation involves the heating of the tanks to a sufficiently high temperature to effect the annealing of the welds. It has been only recently that this matter has been investigated from a metallographic point of view and the results are well worth recording.

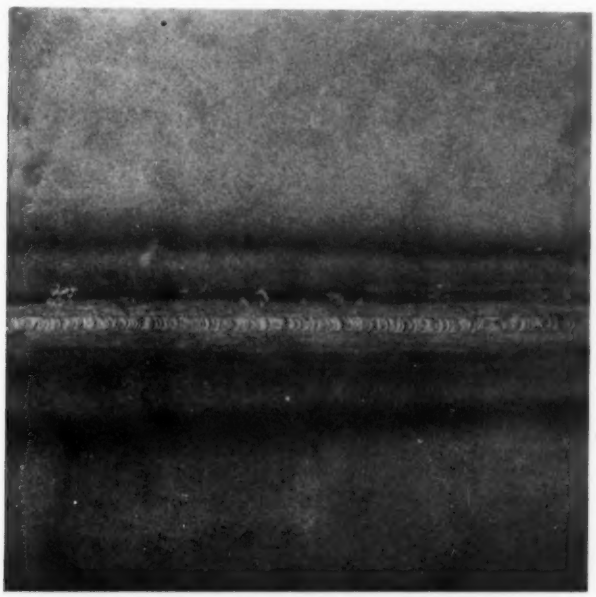

FIG. 3.—Welded plates showing zones of color.

By means of an optical pyrometer, careful measurements have been made as to the actual temperatures reached by the steel in the enameling furnaces. It has been observed that on leaving the furnace the steel temperature ranges from 900 to 950°C (1650 to 1750°F), varying somewhat with different enamels. Referring to the curve indicating the necessary annealing temperature for steels, it will be noted that any steel which would be used for enameling would be at a safe annealing temperature when above

880°C (1620°F). Effort has been made to determine the total time at which a tank undergoing the normal enameling operation would be at a temperature above 900°C (1650°F) and a very reasonable approximation indicates that this time is 15 minutes. From a purely theoretical point of view this should produce a very thorough annealing. Considerable experimental work to support this, however, has been carried on and some typical observations will be reviewed.

FIG. 4.—Structure of plate 10″ from weld.

Two sheets of steel (approximately 0.15% carbon), one foot wide and 2 feet long, were sand blasted and welded together after the usual beveling of the edges. The removal of the mill scale by the sand blast before welding made possible an interesting observation as to the color effect produced by the welding.

Figure 3 shows the weld very distinctly as well as the color effects in the adjacent steel. Starting at the edge of the steel away from the weld, the color is that of sand-blasted steel up to a point approximately 5″ from the center of the weld where the

color works through the usual straw colors up to the deep blue and purple characteristic of steel heated to the corresponding temperatures. The general extent of these influences is obvious from the figure.

The weld was ground smooth on either side and for experimental work several strips 1″ wide were sawed out perpendicular to the weld. Metallographic specimens were prepared covering the entire range of heat conditions.

Figure 4 shows the structure of the metal about 10″ from the weld, obviously well beyond the influence of the heating. It will be noted that the structure is that of a plate worked slightly below its critical temperature as evidenced by the tendency for banding.[1]

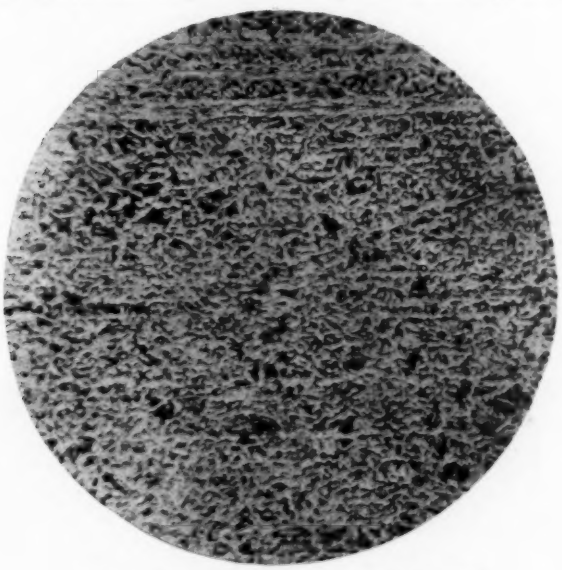

FIG. 5.—Structure of plate 4″ from weld.

Figure 5 was taken at the edge of the straw color, approximately 4″ from the center of the weld. The banded structure is still apparent though possibly slightly less pronounced. Figure 6 was taken at the point of most intense blue color 2 3/4″ from the weld and the banded structure is but slightly noticeable. Figure 7 was

[1] See figure 2—number 16.

FIG. 6.—Structure of plate $2^{3}/_{4}''$ from weld.

FIG. 7.—Structure of plate $1^{1}/_{2}''$ from weld.

taken inside the maximum blue line 1 1/2" from the weld and the banded structure is practically eliminated.

Figure 8 shows the structure of the filler run in between the plates. This filler shows on analysis about 0.05% carbon. It has the normal structure of low carbon iron. Very minute areas of pearlite can be detected here and there.

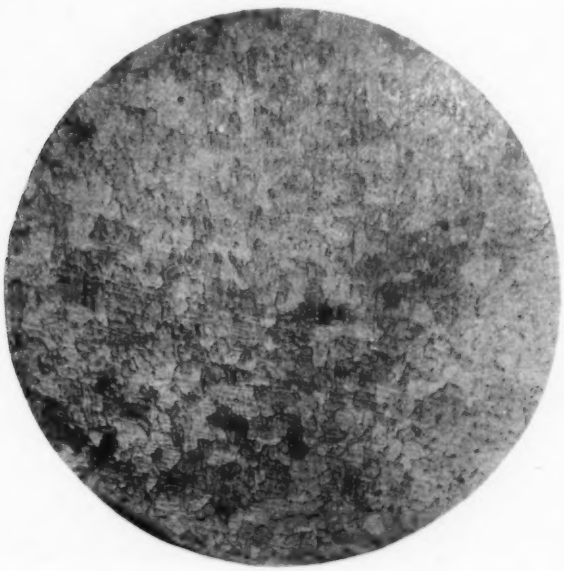

FIG. 8.—Structure of filler.

Figure 9 shows the junction between the plate and the filler. At the extreme right of the figure is seen a structure essentially the same as that of figure 8. The major portion of the figure, however, indicates the coarse irregular structure which has been produced by the intense heating of the plate above its critical temperature. It is interesting to compare this structure with that given as typical of electrically welded sheets on page 12, figure 2, Catalog of the H. M. Kellog Co. This structure also compares very favorably with that shown in figure A, on page 123 of Hoyt's "Principles of Metallography." Hoyt terms this structure "Widmanstattian" and refers to it as "being indicative

of weakness and lack of toughness. It is more commonly found in steel castings and in forgings which have been finished at too high a temperature.....the substitution of a fine grained uniform structure results in an improvement in the mechanical qualities."

FIG. 9.—Junction—unenameled specimen.

Several strips from this sample were enameled in the usual manner following which portions of the weld were examined microscopically. It was found that the structure of the filler was essentially the same as that previously noted while the coarse structure of the plate as shown in figure 9 had been reduced to a fine one of the same general type as that originally existing in the plate, the banded structure being absent.

Figure 10 shows the junction between the filler and the plate indicating a reasonably even distribution of the pearlite among the ferrite grains.

Several observations of the same genreal type have been made both on experimental pieces, and on samples taken from units which had been processed commercially. We will take time to present but one additional case.

FIG. 10.—Junction—Enameled specimen—from same weld as figure 9.

Figure 11 shows the junction between the filler and plate in which the plate structure had taken the form of a very coarse grain. It is interesting to compare this structure with that given by S. W. Miller,[1] who discusses "Some Structures with Steel Fusion Welds," and shows a figure of an oxy-acetylene weld exhibiting the coarsening of grain of ordinary metal.

A specimen from the same weld was enameled and later examined microscopically. The results are shown in figure 12, in which the coarse structure of figure 11 has been reduced to a very fine structure with reasonably uniform distribution of pearlite.

[1] See figure 2, *Trans. Amer. Inst. Min. Eng.*, **1918**, 339.

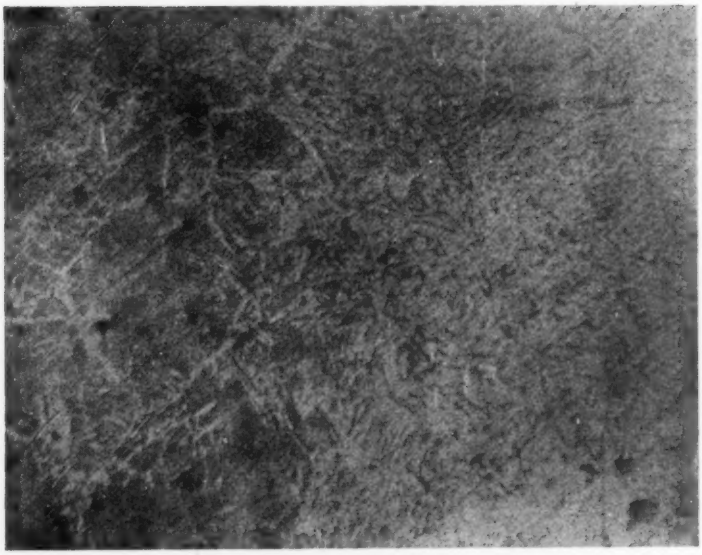

FIG. 11.—Junction—unenameled.

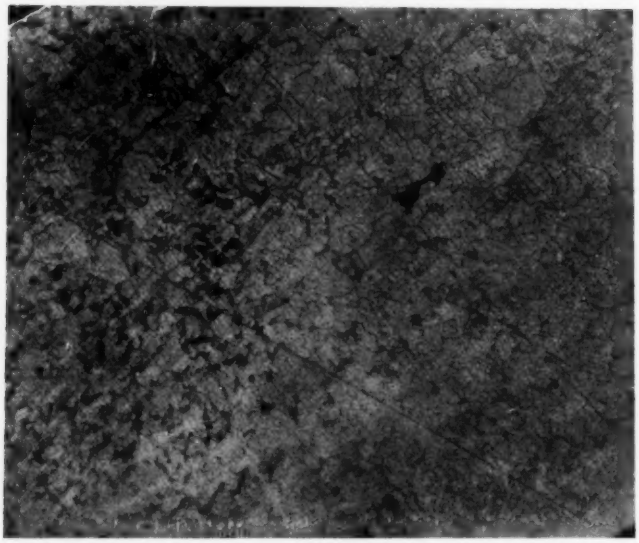

FIG. 12.—Junction—enameled from same weld as figure 11.

The above work indicates very definitely that the enameling operation furnishes very thorough annealing of the welds producing structures which indicate very desirable mechanical qualities as compared with those suggested by the unenameled samples and often considered as characteristic of oxy-acetylene welds.

This work will be followed by physical tests which should throw considerable light on the practical results of the changes noted herein.

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THE SEPARATION OF LIME FROM DOLOMITE[1]

By H. G. Schurecht

ABSTRACT

Calcination.—Magne ium carbonate in dolomite is decomposed by calcining for one hour at 800°C. Calcium carbonate is not completely decomposed until after calcination at 960°–1040°C for one hour.

Treatment with sulphuric acid.—By adding sufficient H_2SO_4 to milk of dolomite to react with the lime present, a bulky precipitate of $Mg(OH)_2$ is formed which may be partially separated from the finer $CaSO_4$ by screening through a 120 mesh sieve. The residue on the screen contains about 68.3 per cent MgO and represents over 50 per cent of the original CaMgO.

Flotation.—The best results by flotation were obtained by removing the fine material from the raw dolomite and then calcining at 920°C. Wood creosote flotat'on oil number 400 was found best suited for this separation. The concentrates removed by flotation, however, represent only 25 per cent of the dolomite originally treated.

Leaching and screening.—By a leaching and screening treatment it is possible to obtain a product containing about 80 per cent MgO. This is superior to Canadian magnesite in MgO content.

Elutriation.—By an elutriation treatment it is possible to obtain a residue containing over 85 per cent MgO and representing about 30 per cent of the original dolomite.

Introduction

In previous work[2] the writer found that it was difficult to produce fire brick from calcined mixtures of dolomite and hematite, which would withstand storage in the open air for periods of time longer than six months. During this period the free lime and lime compounds combine with the moisture or change in volume causing the brick to disintegrate. Finer grinding of the raw dolomite before calcination with hematite proved beneficial in many cases but did not entirely overcome the tendency to disintegrate. Calcining the raw dolomite and hematite mixtures at higher

[1] Received March 14, 1921. Published by permission of the Director, U. S. Bureau of Mines.

[2] This Journal, **4** (1921).

temperatures in some cases increased the tendency of the resultant material to disintegrate. Magnesite on the other hand, can be easily dead-burned and magnesite brick withstand storage in open air, remaining sound, for long periods of time. Magnesite brick are also more resistant to slagging action than dolomite brick and for this reason it has been claimed[1] that the life time of a magnesite bottom is three times that of a dolomite bottom.

Prior to the war, practically all the magnesite used by the steel industries came from Austria and was laid down on the American seaboard at from $15 to $18 per ton. During the war[2] the prices of domestic magnesite increased to $50, $75 and finally $100 per ton the chief source of this material being the State of Washington.

At the present time, caustic lump magnesite is quoted[3] at $35 to $40 per ton, carload lots, f.o.b. California. In the Chicago district it is quoted at $57.70 and at the Atlantic seaboard at $61 and $63. Dead-burned magnesite is quoted at $38 per ton at Chewelah, Washington and $58–$64 per ton at Chester, Penna.

Dolomite,[4] 1 and 2 man size, is quoted at $1.60–$1.65 per ton. 2–8 in dolomite is quoted at $1.55–$1.65 per ton f.o.b. Plymouth Meeting, Pa.

Since the price of one ton of dead-burned magnesite delivered to Pennsylvania points is at present equal to that of 37–39 tons of raw dolomite, the problem of concentrating the magnesia in dolomite by reducing the lime content, was considered worthy of investigation.

The work herein reported is therefore limited to a study of the separation of lime from dolomite. Various processes already patented were studied as well as other processes which were developed in the laboratory as the work progressed.

The Work of Previous Investigators

Closson[5] separated magnesia from dolomite by adding 8750 parts of magnesium chloride to 1250 parts of caustic dolomite.

[1] McDowell, Spotts, J., and Howe, R. M., "Magnesite Refractories," THIS JOURNAL, **3**, 189 (1920.)

[2] Bankson, R. A., "Glory Holes and Magnesite," *Eng. & Min. Jr.*, **111**, 49–52 (1921.)

[3] The Market Report, *Eng. & Min. Jr.*, **111**, 85 (1921).

[4] *Eng. & Min. Jr., loc. cit.*

[5] This appears as footnote 1 on p. 560.

The lime in the dolomite combines with the nagnesium chloride forming soluble calcium chloride and a precipitate of magnesium hydroxide. The soluble calcium chloride is then removed by filter pressing leaving 1000 parts of magnesia in the press.

By the Pattinson method[2] magnesia is separated from dolomite by subjecting the same to a pressure of 5–6 atmospheres in the presence of carbonic acid. The solution is then heated with steam and the precipitate of magnesia dried.[3]

In another process[4] dolomite is calcined at 500–600°C at which temperature the carbon dioxide is driven from the magnesium carbonate, while the calcium carbonate remains in the undissociated form. The magnesia is then removed either by air or water separation.

By the addition of a small amount of sugar or molasses to milk of dolomite, the lime readily goes into solution while magnesium hydroxide remains as a precipitate. Scheibler[5] uses this reaction in the separation of lime from dolomite. He adds 10–15 per cent sugar or molasses solution to milk of dolomite. The solution of the lime by sugar takes place rapidly leaving magnesium hydroxide as a precipitate. The lime sugar solution is then separated by decantation or filtration. The sugar and lime are recovered by passing CO_2 through the solution thus forming CaCO and sugar.

Bradburn[6] uses a by-product in the manufacture of soda by the ammonia process to separate magnesia from dolomite. This by-product contains 60–90 parts NaCl and 120–180 parts NH_4Cl. The dolomite is so calcined to decompose both the calcium and magnesium carbonates. The calcined dolomite is then hydrated. The milk of dolomite is then mixed with the solution of sodium and ammonium chlorides. The liquor contains calcium chloride, magnesium chloride and sodium chloride with an excess of NH_4Cl.

[1] Scherer, R., *Der Magnesit*, A. Hastleben's Verlag, Wien und Leipzig, **84**, (1908).

[2] *Der Magnesit, loc. cit.*, p. 134.

[3] This is substantially a literal translation but does not describe the process clearly.

[4] *Der Magnesit, loc. cit.*, p. 134.

[5] *Ibid.*, p. 135.

[6] Bradburn, J. A., "Process of Obtaining Magnesia." U. S. 1,156,662, Oct. 2, 1915.

The clear liquor is decanted and sufficient milk of dolomite is added to convert the $MgCl_2$ into $Mg(OH)_2$.

The $Mg(OH)_2$ from the milk of dolomite does not enter in the reaction but is collected with the precipitated $Mg(OH)_2$. The mixture is then passed through a settling tank and the heavy MgO is deposited near the inlet while the light $Mg(OH)_2$ together with the $CaCl_2$ is carried off in the overflow. The $Mg(OH)_2$ is then filtered and washed free from chlorides.

Another process[1] consists in crushing calcined dolomite to 8–40 mesh size and subjecting it to a leaching process with cold water which dissolves the lime leaving the magnesia as an insoluble residue. This process is continued until the residue contains not more than 3–10% lime.

A patent was issued to Mitchell[2] for obtaining magnesium oxide from dolomite by calcining at 950°F which decomposes the magnesium carbonate but not the calcium carbonate. The magnesium hydroxide formed on the addition of water is separated by running water or air (this process being practically the same as that described under reference No. 9).

Epsom salts[3] are made from dolomite by treating dolomite with sufficient H_2SO_4 to react with the calcium and magnesium present. The resultant magnesium sulphate solution is then filtered from the calcium precipitate.

Experimental Methods

The following methods are briefly outlined for the processes used for the separation of lime from dolomite in this work. In each case chemical analyses were made of the samples separated and the CaO and MgO contents expressed in terms of the oxides. This was also done when undecomposed $CaCO_3$ was present to make the results more comparable.

[1] "Refractory material and process for making same," U. S. 1,270,819, July 2, 1918.

[2] Mitchell, A. M., "Process for Obtaining Magnesium Oxide from Dolomite," U. S. 1,273,110, July 16, 1918.

[3] Newth, G. S., "A Text Book of Inorganic Chemistry," Longmans, Green & Co., New York, 1905, 575.

1. Treatment with Sulphuric Acid.—1000 Kilogram samples of dolomite were calcined at 1040°C and hydrated. Enough H_2SO_4 was added to react with the $Ca(OH)_2$ and the fine crystals of $CaSO_4$ thus formed were removed from the bulkier $Mg(OH)_2$ by screening.

2. Separation by Flotation.—1000 Kilogram samples of dolomite crushed to 8–20 mesh size were calcined at 760°, 840°, 920° and 1040°C and hydrated. The magnesium hydroxide was removed to some extent by flotation. This was possible because of the fact that $Mg(OH)_2$ is more colloidal (slimy) than the undecomposed calcium carbonate and certain forms of lime hydrate.

The same experiments were made on dolomite which had been screened through a 100 mesh sieve, previous to calcination.

Wood creosote flotation oil number 400 of the Pensacola Tar & Turpentine Co. was found best suited for this separation and therefore was used.

3. Separation by Leaching and Screening.—1000 Kilogram samples of dolomite crushed to 8–20 mesh size were calcined at 840°, 920°, and 1040°C and hydrated. Different samples of each were then passed through the following mesh size screens: 4, 20, 65, 100 and 200. The materials passing through the screens were then leached with water for 4 days. The same experiments were made with dolomite which had been screened through a 100 mesh sieve previous to calcination.

4. Elutriation Tests.—1000 Kilogram samples of dolomite crushed to 8–20 mesh size were calcined at 840°, 920°, and 1040°C and hydrated. Sieve analyses were made to separate the hydrated material into the following sizes: 20, 20–65, 65–100, and 100–200 mesh. The material which passed through 200 mesh was separated by elutriation according to the method described for the elutriation of clays.[1] The same experiments were made with dolomite which had been screened through a 100 mesh sieve previous to calcination.

Results

The Effects of Calcination Temperatures on the Dissociation

[1] THIS JOURNAL, **3**, 355–78 (1920).

of Dolomite.—Cedarville dolomite[1] crushed to 8–20 mesh was calcined for 1 hour at 720°, 760°, 800°, 840°, 880°, 920°, 960°, 1000° and 1040°C, respectively. The same was repeated with dolomite screened through a 100 mesh sieve. The losses in weight at these temperatures are shown in figure 1.

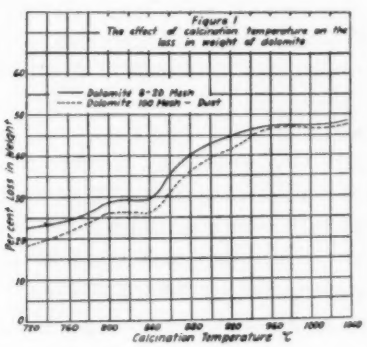
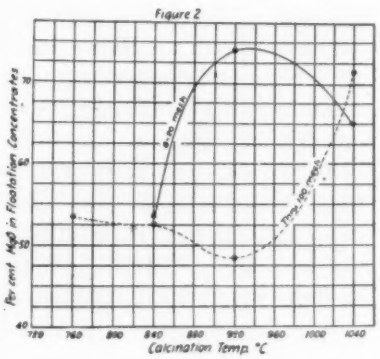


Figure 1
The effect of calcination temperature on the loss in weight of dolomite
Dolomite 8-20 Mesh
Dolomite 100 Mesh - Dust
Per cent loss in weight
Calcination Temperature °C



It is obvious that 8–20 mesh dolomite may be calcined more thoroughly than the 100 mesh size as shown by the greater loss in weight of the coarser sizes.

The magnesium carbonate is apparently decomposed by calcining at 800°C while the calcium carbonate is not completely decomposed until 960°–1040°C.

Treatment with Sulphuric Acid.—In treating the hydrated dolomite with sulphuric acid, sufficient acid was added to the milk of dolomite to react with the lime present. A bulky precipitate of $Mg(OH)_2$ is thus formed and the calcium sulphate is present as extremely fine needle-like crystals. Due to the difference in the structures of the $CaSO_4$ and $Mg(OH)_2$, the former may be removed largely by screening through a 120 mesh sieve. The residue remaining on the screen contains about 68.3% MgO and, in terms of $CaMgO_2$, represents over 50% of the dolomite originally used. By violent agitation and washing with water all of the colloidal

[1] The chemical analysis of Cedarville dolomite is as follows: CaO=30.2 %, MgO=21.8%, CO_2=46.8%, SiO_2=0.4%, Al_2O_3=0.5%, Fe_2O_3=0.5% and hydro. water=0.1%.

$Mg(OH)_2$ may be forced through the sieve. Utilization of the calcium sulphate thus removed in the manufacture of plaster of paris or Estrich's plaster is a possibility, though not investigated at this time.

Separation by Flotation.—Figure 2 shows the results obtained in the separation of magnesium hydroxide from dolomite by flotation. The best results were obtained by calcining 8–20 mesh dolomite to 920°C. Removing the fine material through a 100 mesh sieve before calcination seems necessary as shown in tests made on dolomite which had been screened through a 100 mesh sieve. For example, the concentrates from dolomite which had been crushed to 8–20 mesh size and calcined at 920°C contained 74 per cent MgO, whereas that from dolomite screened thru a 100 mesh sieve and calcined at the same temperature contained only 48.5 per cent MgO. This is due largely to the fact that the fine undecomposed calcium carbonate is carried away with the froth.

When calcined at 1040°C the concentrates obtained from the 8–20 mesh and 100 dust dolomite, were both comparatively high in MgO, containing over 65 per cent. This may be partly due to the leaching action of water on the caustic lime present.

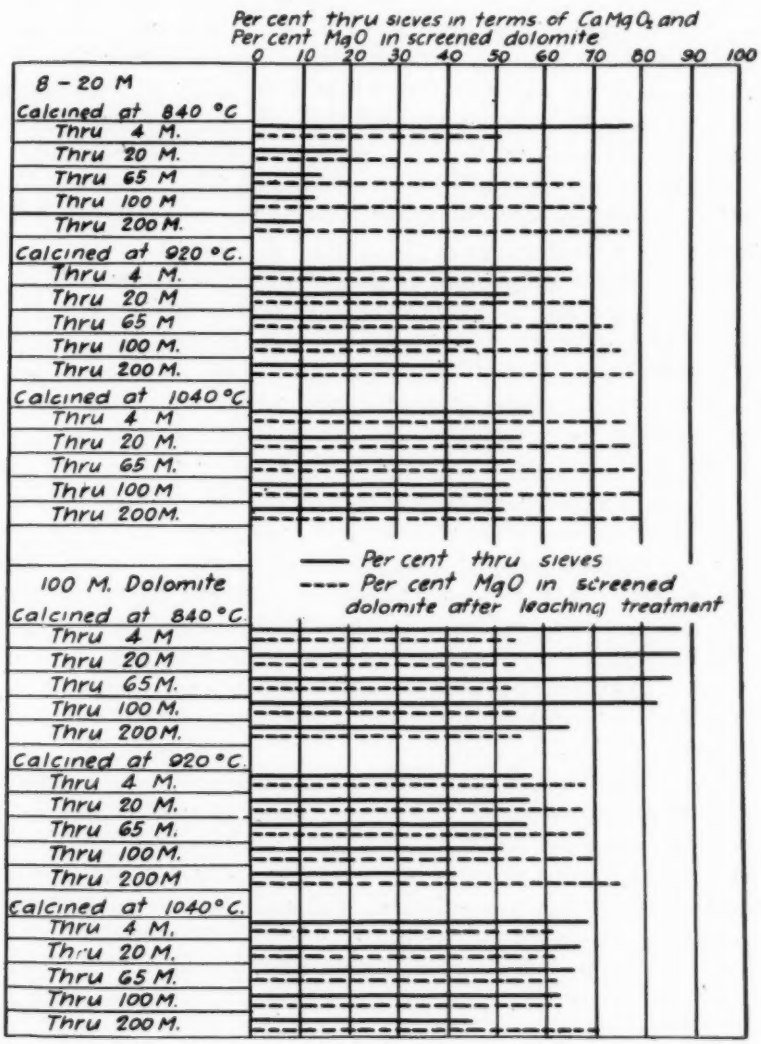
The concentrates removed by flotation, however, represent, in terms of $CaMgO_2$, only about 25 per cent of the original dolomite used. By the sulphuric acid and screening and leaching treatments, a 50 per cent recovery is obtained. If more than 25 per cent concentrate is forced over the flotation cell, it rapidly increases in lime content.

Separation by Leaching and Screening.—The solubility of $Ca(OH)_2$ is 0.185 gram per 100 grams of water (Seidell), whereas that for $Mg(OH)_2$ is 0.014 gram per liter (Riddell),[1] at room temperatures. By allowing water to run for a number of days over dolomite, calcined sufficiently high to decompose both the $MgCO_3$ and $CaCO_3$, a large per cent of the lime may be removed owing to its greater solubility in cold water and to its finer particles which are carried away mechanically.

[1] Duschak, L. H., "The Determination of Free Calcium Oxide in Caustic Burned Magnesium Oxide." *Bureau of Mines Reports of Invest.*, June, 1–2, 1920.

In this portion of the work therefore, the effect of size of grain and calcination temperature on the separation of lime from dolomite by leaching and screening were studied. Water at room

Figure 3



temperatures was allowed to run over 300-gram samples of calcined dolomite at the rate of 100 cc. per minute for 4 days. The undecomposed calcium carbonate was then further removed by screening.

The results are shown in figure 3. It is obvious that the best results are obtained by crushing dolomite to 8–20 mesh, calcining at 1040°C, and allowing water to flow over it for 3 to 4 days. For example, by crushing dolomite to 8–20 mesh size, calcining at 1040° hydrating with water, screening through a 100 mesh sieve and leaching for 3 to 4 days, a product is obtained containing about 80 per cent MgO with less than 3 per cent $SiO_2+Al_2O_3+Fe_2O_3$ This material represents, in terms of the oxides, about 50 per cent by weight of the dolomite originally used showing that practically all of the MgO is recovered from the dolomite by this method. Calcined Canadian magnesite contains 72.7% MgO and 6.7% $SiO_2+Al_2O_3+Fe_2O_3$.[1] Obviously by this process, a product richer in magnesia than Canadian magnesite may be produced. Screening the dolomite through a 100 or 200 mesh sieve after hydration is advantageous especially if undecomposed calcium carbonate is present as is shown in figure 3.

The results also show that it is advisable to remove material finer than 100 mesh before calcination. This is partly due to the fact that the finer undecomposed calcium compound passes through the screens in the subsequent screening thereby increasing the lime content in the material passing the screens. Also the fine dolomite dust is not as thoroughly decomposed as is the coarser material due to freer egress of the CO_2 through the voids in the coarse material. The percentage of undissociated dolomite is therefore higher in the fine material.

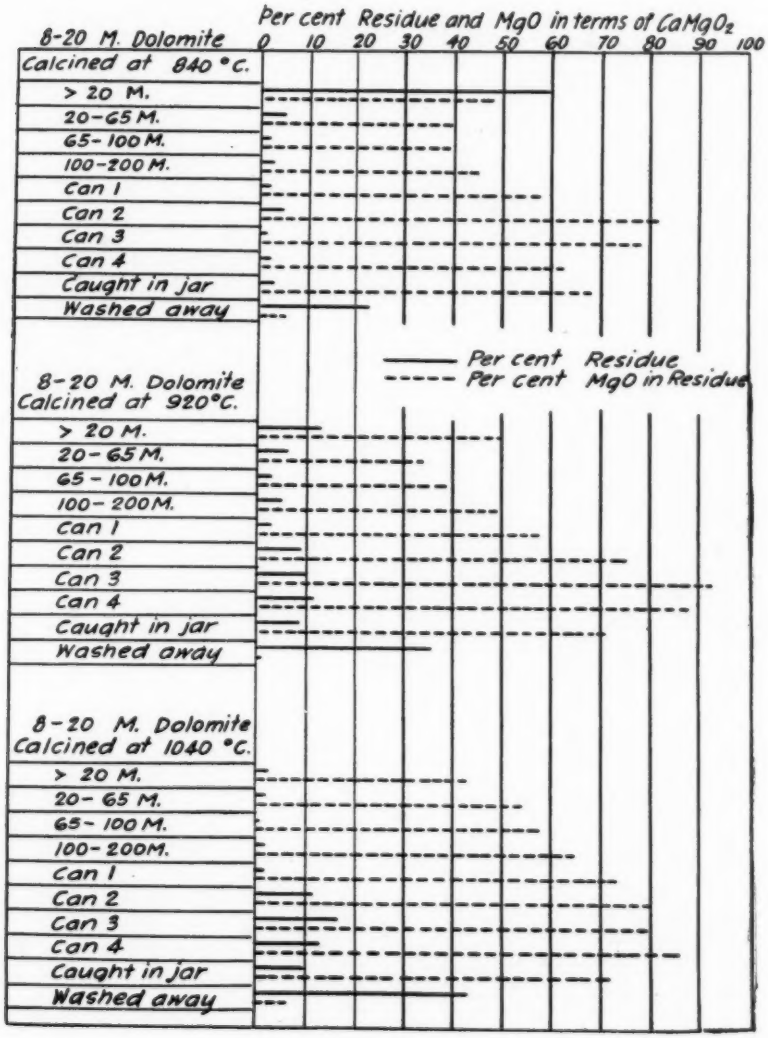
Elutriation Tests.—The results of elutriation tests on dolomite are shown in figures 4 and 5. In most cases the percentage MgO in the portions separated increases up to can 3 and then decreases in can 4 and in the overflow jar. As high as 44 per cent lime is washed away being largely dissolved in the water. The velocities of flow in the respective cans are as follows:

	Can. 1	Can 2	Can 3	Can 4	Per sec.,
Velocity.....	0.347	0.180	0.059	0.0197	mm.

[1] Analysis made by A. Fieldner, U. S. Bureau of Mines, Pittsburgh, Pa.

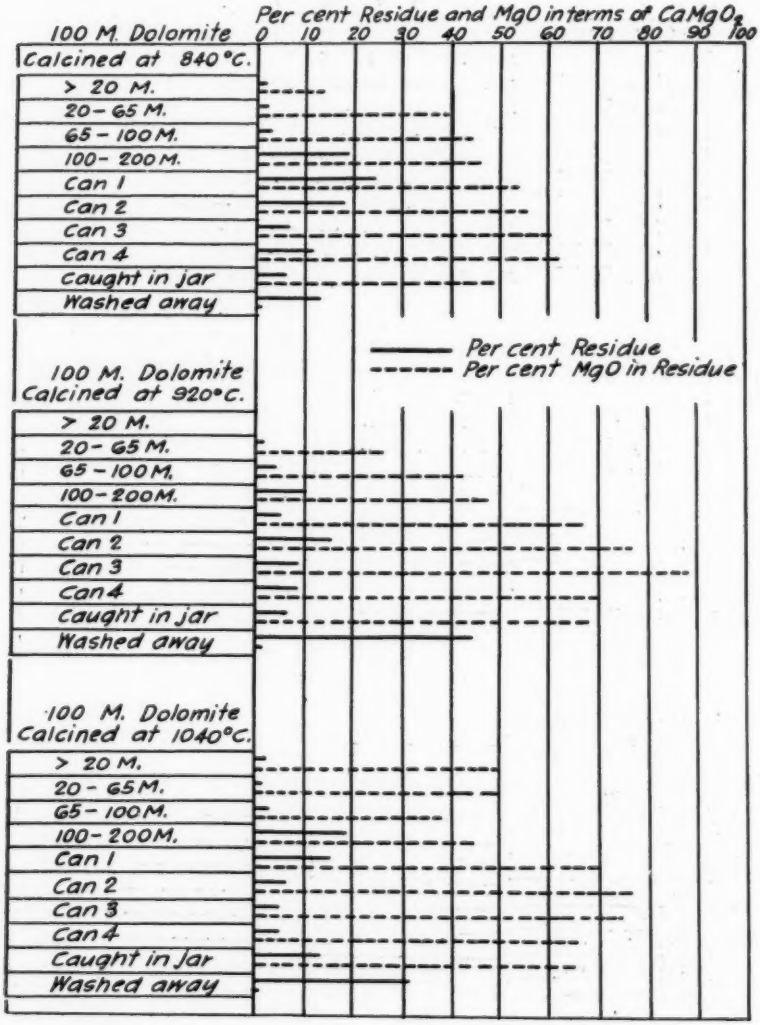
When 8–20 mesh dolomite was calcined at 920 °C and elutriated, the material deposited in can 3 contained 92% MgO. This however, only represents 9.5 per cent of the dolomite originally treated in terms of oxides or 21 per cent of the MgO originally

Figure 4



present in the dolomite. By combining the residues deposited in cans 2, 3 and 4, a product averaging 85.4 per cent MgO was obtained which represents 30 per cent of the dolomite originally used in terms of the oxides or 61.6 per cent of the MgO present in the

Figure 5



original dolomite sample. In other words, by using an elutriation system whereby the velocity of flow in vessel 1 is 0.347 mm. per sec. and in vessel 2 is 0.0197 mm. per sec., a product containing 85.4 per cent MgO is deposited in can 2 which represents 61.6 per cent of the MgO present in the dolomite originally treated.

Conclusions

The leaching and screening process gives the most satisfactory yields of MgO rich material.

If a high degree of purity is desired, combinations of the above treatments may be used, but for many purposes a product containing over 80 per cent MgO would probably be sufficient.

These processes have not yet been tried on a large scale and the results above are those obtained in the laboratory.

In conclusion the author wishes to acknowledge his indebtedness to Mr. R. T. Stull for many valuable suggestions in the work.

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PLASTIC MAGNESIA CEMENTS[1]

By P. H. Bates and Roy N. Young

ABSTRACT

Preparation of the Cement.—Crystalline magnesite from Washington was crushed to three sizes—to pass a No. 6 sieve but retained on a No. 10; between a No. 10 and a No. 30 and to pass a No. 60 sieve. These sizes were burned separately in an electric rotary kiln at 600°, 650°, 700°, 800°, and 900°C.

Tests of the Cement.—The cement was tested in three flooring mixes in which the MgO was 35.0, 42.5 or 50.0% by weight, and in three stucco mixes in which the MgO was 11, 22 or 33% by weight. Tensile and compressive strength specimens were broken at 24 hours, 7 and 28 days. The coefficient of expansion was determined at 48 hours, 4, 7, 28 and 90 days. Other properties determined were time of set, consistency, soundness, fineness and effect of density of the chloride solution used.

Results.—The property of the calcined cement is materially affected by the size of ore, temperature and duration of burning. The rate of reaction of MgO with chloride materially decreases with increased temperature of burning.

Decreasing the concentration of the magnesium chloride solution (down to 22° Bé) accelerates the set of freshly calcined magnesite and retards the set of magnesia which has become hydrated to a considerable extent through exposure. Exposure to moisture before using as well as increasing the consistency of the mixture lengthens the setting lime.

A composition which expands excessively and warps or buckles is not necessarily "unsound," but one which disintegrates within a comparatively short time may be considered as such. Used in this sense, "unsoundness" is believed to result from the presence of free magnesia which hydrates after the mixture has hardened, and not from the presence of lime. Therefore, soundness is not a property of the magnesia alone but depends upon the extent to which it reacts with the magnesium chloride, water, or carbon dioxide before hardening takes place and upon the amount of hydration which subsequently occurs. The steam or cold water tests are unsatisfactory as accelerated tests for soundness of magnesia mixtures.

Under the conditions of these tests, the best material with regard to setting time and strength was produced at a temperature of 800°C. However, the magnesia burned at 650°C and which gave comparatively very low strength when gaged with a 22° Bé solution of magnesium chloride, gave excellent strengths when gaged with more concentrated solutions. Materials tested with 22° Bé solution and giving satisfactory results would not necessarily

[1]Received April 15, 1921. Published by permission of the Director, U. S. Bureau of Standards.

be satisfactory with a higher or lower concentration. It seems that general specifications should also include limits corresponding with tests in which higher and lower concentrations are used.

No relation was found between the volume change and any other property of the magnesia. Tests of any particular mixture are no indication of the behavior of the same magnesia in other mixtures. However, the laboratory tests indicate that the leaner mixtures undergo less change in volume than the richer ones. Furthermore, a number of strict comparisons between laboratory and field tests indicate that the laboratory tests of volume changes as made are no index to the behavior of the material under actual service.

The lean mortar mixture proved to be the most suitable of any used for testing magnesia, and in this case the tensile strength furnished as much, if not more, information than the compressive strength.

Introduction

The data presented and discussed herewith represent the results obtained from a preliminary investigation dealing with the production of plastic (or so-called caustic) magnesia from domestic magnesites and the testing of the product to determine some of its properties as a cementing medium when mixed with a solution of magnesium chloride and certain inert fillers, as finely ground sand, asbestos, wood flour, and coloring.

Previous to the late war practically all of the magnesia used as a cementing agent for resilient floors came into this country from Greece. Usually it had been calcined before its arrival and in the majority of these cases the calcination had been made in Holland for the purpose of obtaining carbonic acid. The cutting off of this supply necessitated the more active development of domestic sources of similar material. These had been noted to exist in California and had been worked for some time for a number of purposes other than the production of plastics for flooring or stucco. Like the Grecian ore or a similar ore from Venezuela the California ore is said to be amorphous, (and microscopically it does appear so), and is in striking contrast to the magnesite from the State of Washington, which is very crystalline.

While a very satisfactory ore was therefore at hand, there was but little known in regard to its proper preparation and calcining as a plastic cement, or of the properties which the latter should have to assure satisfactory results in its application. An investigation was therefore undertaken to determine these factors

using the amorphous ore. Later it was thought desirable to include the crystalline variety on account of the very large deposits in Washington and also because it was learned that some of this variety from Austria had been going into the plastic trade abroad.

The complete outline of the investigation includes the burning of various sized ores from different localities at different temperatures. From the ground calcine the usual tensile, compressive and transverse strength test pieces will be made, as well as tests to determine the constancy of volume of different flooring and stucco mixtures. There will also be made a number of test panels of flooring and stucco, which will be subjected to actual service conditions.

This paper, however, covers only the results of some preliminary work in which one variety of magnesite alone was investigated and in which the prime object was to determine how the ore crushed to different sizes would be affected by burning at different temperatures. It would in a measure determine the sizes and temperatures to be used in the remainder of the work. On account of its nature only part of the tests outlined above were carried out. The results though not conclusive are so suggestive of different lines of future work, that it was thought they would be of much interest to those engaged in the investigation of oxy-chloride cements.

Description and Properties of Materials

Magnesia.—The magnesite used throughout these tests was from Stevens County, Washington, and all of the same shipment except as shown in table I. It was crushed to three different sizes. The first contained the particles which passed a No. 6 sieve and were retained on a No. 10 sieve, the second was between the No. 10 and No. 30 sieves, and the third was sufficiently fine to pass a No. 60 sieve. Each size was burned at the temperatures 600°, 650°, 700°, 800° and 900°C making 15 burns in all. The burning was done in a small electric resistance rotary kiln. During the operation of the kiln, the variation from the desired temperature was seldom more than 10° as found by temperature readings taken every 10 minutes. The time required for the material to pass

through the kiln was approximately one hour. The material which was crushed to the finest size progressed somewhat slower than the coarse size, and at the higher temperatures had a slight tendency to stick to the kiln lining. After burning the material was ground and placed in tightly covered metal containers until used.

The results of the chemical and physical tests of the raw and finished materials are given in tables I and II, respectively. Attention is called to the range of CO_2 content, and the variation in the amounts of this constituent with the fineness of ore and the burning temperature. It may be well to explain that these analyses were made after the burned material was ground, which

TABLE I—RESULTS OF CHEMICAL ANALYSES OF THE RAW AND FINISHED MATERIALS

Lot	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	CO_2	Ig. loss
RAW MATERIALS							
1	1.03	.80	.52	1.95	46.22		49.81
2	2.12	.72	.66	.88	45.18		49.96
BURNED MATERIALS							
Burn	Lot						
xB-1*	2					28.00	30.50
B-2*	2					7.05	17.50
B-3*	1					5.60	9.92
xC-1	2					4.35	7.00
C-2	2					3.50	6.90
C-3	2					3.80	5.67
xD-1	2					2.85	4.00
D-2	2					2.65	3.40
D-3	2					1.85	2.55
xE-1	2					1.30	2.42
E-2	2					1.75	2.40
E-3	2					.85	1.75
xF-1	2					.20	.75
F-2	2					.15	.70
F-3	2	4.49	1.36	1.68	1.57	90.40	.28	.50

xB burns made at 600° *1 Material sized between No. 6 & No. 10 sieves
C " " " 650° 2 " " " No. 10 & No. 30 "
D " " " 700° 3 " all through No. 60 "
E " " " 800°
F " " " 900°

TABLE II—RESULTS OF PHYSICAL TESTS ON THE FINISHED BURNED MATERIALS

CEMENT	FINENESS Per cent retained on No. 100 sieve	No. 200 sieve	(SPECIFIC GRAVITY)	LOOSE WT. PER CU. FT. (LBS.)
B-1	0.2	12.8	3.05	60.7
B-2	0.4	9.6	3.06	46.8
B-3	0.6	12.6	3.13	45.5
C-1	0.6	10.8	3.22	42.8
C-2	1.4	13.1	3.26	45.6
C-3	0.2	9.7	3.05	43.5
D-1	1.4	12.6	3.05	50.2
D-2	2.4	12.6	3.13	52.8
D-3	0.8	10.1	3.22	51.7
E-1	1.8	12.8	3.34	52.3
E-2	2.2	14.1	3.38	52.8
E-3	1.4	14.3	3.35	51.4
F-1	0.9	12.3	3.40	50.9
F-2	1.9	13.6	3.43	53.2
F-3	0.5	10.3	3.44	55.5

will, to some extent, account for the amount of water present. In this connection, it is interesting to note the relative activity of the different burns towards moisture as shown by the following tests.

Five grams of the finished material from each burn made on the coarsely crushed magnesite were spread out on a cover glass and placed in moist air which was free from carbon dioxide. At certain periods the samples were dried and weighed, and the increase in weight was converted to percentage increase in combined water. These data are given in table III. It may be seen that the

TABLE III—PERCENTAGE INCREASE IN WEIGHT DUE TO HYDRATION OF MAGNESIA FROM VARIOUS BURNS UPON EXPOSURE TO MOIST ATMOSPHERE

Burn	Duration of exposure				
	1 Day	2 Days	7 Days	14 Days	28 Days
B-1	8.68	10.14	16.88	17.08	
C-1	13.37	19.43	24.95	26.22	
D-1	10.50	14.55	25.55	27.55	
E-1	1.44	1.84	11.76	21.56	21.96
F-1	0.97	2.24	10.13	15.30	16.81

activity of the magnesia toward moisture decreased as the calcining temperature increased. Although the amount of hydration of burn B-1 was less than that of the others, due to the large amount of undecomposed $MgCO_3$, yet if calculated on the basis of caustic magnesia content it was higher.

Magnesium Chloride.—The results of analysis of the magnesium chloride solution were as follows:

SiO_2	0.0007%		
R_2O_3	.0024		
SO_3	.0085		
CaO	.085		
MgO	8.80	=	20.86% $MgCl_2$
Cl_2	15.50	=	20.83% $MgCl_2$
Specific gravity	1.184		

Flint.—This material is that known as "potters' flint." The lot which was used in these tests had a fineness of 99 per cent through a No. 100 sieve and 89.6 per cent through a No. 200 sieve.

Asbestos.—The asbestos used in the flooring mixtures was what is known commercially as grade "C."

Details of Tests of Cement Mixtures

Mixtures

Cement from each burn was used in 6 different mixtures of the following compositions.

	Compositions (percentage by weight)			
Marked	MgO	Flint	Sand	Asbestos
AF	50.0	30.0		20.0
BF	42.5	34.5		23.0
CF	35.0	39.0		26.0
AM	33		67	
BM	22	11	67	
CM	11	22	67	

The three containing asbestos will be referred to as flooring (F) mixtures and the others as mortar (M) mixtures.

Mixing

The dry materials were thoroughly mixed before gaging with the magnesium chloride solution and only a sufficient amount

was used at one time for molding 3 to 6 briquettes or 1 to 3 of the larger test specimens. The mixing was done in accordance with the method described in the U. S. Government Specification for Portland Cement. Exceptions to this occurred in a number of cases in which it was impossible to give the full time to the mixing operation on account of the very quick setting property of the material. However, by very rapid work, thorough mixing was accomplished.

Consistency

A standard consistency for the flooring mixtures was adopted, which appeared to be about the same as that used in the testing

TABLE IV—RATIOS OF MAGNESIUM CHLORIDE TO CEMENT AND CONSISTENCIES USED IN THE VARIOUS TESTS

	Flooring Mixtures						Mortar Mixtures					
	** Ratio $\frac{MgCl_2}{MgO}$			*Consistency (% by weight)			Ratio $\frac{MgCl_2}{MgO}$			Consistency (% by weight)		
Burn	A	B	C	A	B	C	A	B	C	A	B	C
B-1	.256	.275	.334	62	58.7	58.7	.119	.150	.262	19	16	14
B-2	.297	.330	.395	72	68	67	.156	.197	.318	25	21	17
B-3	.297	.330	.395	72	68	67	.162	.197	.318	26	21	17
C-1	.297	.330	.395	72	68	67	.168	.206	.337	27	22	18
C-2	.297	.330	.395	72	68	67	.168	.206	.337	27	22	18
C-3	.297	.330	.395	72	68	67	.159	.197	.318	25.5	21	17
D-1	.297	.330	.395	72	68	67	.168	.201	.318	27	21.5	17
D-2	.297	.330	.395	72	68	67	.168	.201	.318	27	21.5	17
D-3	.297	.330	.395	72	68	67	.156	.197	.318	25	21	17
E-1	.297	.330	.395	72	68	67	.144	.178	.300	23	19	16
E-2	.297	.330	.395	72	68	67	.144	.178	.300	23	19	16
E-3	.297	.330	.395	72	68	67	.144	.178	.300	23	19	16
F-1	.297	.330	.395	72	68	67	.125	.169	.300	20	18	16
F-2	.297	.330	.395	72	68	67	.125	.169	.281	20	18	15
F-3	.297	.330	.395	72	68	67	.119	.159	.281	19	17	15

*Per cent (22° Bé $MgCl_2$) of weight of dry mix.

**Ratio $\frac{MgCl_2}{MgO}$ = ratio per cent anhydrous $MgCl_2$ to per cent calcined magnesite and hence is *not* a molecular ratio. In a formula such as as $MgCl_2$. 5MgO the ratio of per cent $MgCl_2$ to per cent MgO is .473.

of neat Portland cement. By an examination of table IV, it may be seen that the same percentage of solution was used for all the flooring mixes of the same proportions with the exception of those which contained magnesia from burn B-1. It is not claimed that this percentage of solution produced the same consistency throughout, but in consideration of the difference in the properties of the various mixes, especially setting time and smoothness, it is believed that this method was more satisfactory than attempting to change the percentage of solution slightly in each case as might have been done had the judgment of the operator been relied upon entirely in duplicating consistencies.

In the mortar tests it was necessary to follow the latter procedure and here again it was difficult to decide just what was the proper consistency for the reasons mentioned above. It was impracticable to use any measuring device on account of the rapid stiffening of the mixture which occurred in many cases. No doubt some method such as is used in determining consistency or "flowability" of cement or concrete may be applied to compositions of these types, assuming that quick setting or very early stiffening would preclude the use of such material in practice.

Time of Setting

The time of setting was determined by the use of Gilmore needles. No difficulty was encountered in applying this method to the flooring mixtures, but the mortars required a slight change in the manner of observing the set. As the condition of the latter mixtures approached the initial set the penetration of the needle became very ununiform, for if the tests were made at different places on the same specimen the needle made no impression in some places and penetrated appreciably in others. This difficulty was overcome as follows: It was found that after the needle ceased to penetrate there was usually a short time during which the material yielded under the pressure of the needle. It appeared that this yielding was not affected by the coarse sand particles as was the penetration of the needle. It was therefore considered that a mortar had attained its set when the material ceased to yield under application of the needle, and in this way perfectly satisfactory results were obtained.

TABLE V—SETTING TIME

	Flooring Mixtures						Mortar Mixtures					
	Initial			Final			Initial			Final		
	*A	*B	*C	A	B	C	A	B	C	A	B	C
	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.	h. m.
B-1	35	35	1 5	2 40	2 55	3 15	3	5	15	5	15	25
B-2	10	15	20	40	1 30	1 40	2	2	5	3	5	20
B-3	40	40	1 10	2 15	2 25	1 45	5	5	15	10	15	35
C-1	45	55	40	1 40	1 55	2 30	10	10	35	30	50	1 10
C-2	20	25	40	1 25	1 5	2 20	5	10	15	10	20	45
C-3	1 10	1 30	1 30	3 10	2 45	3 10	30	1	1 10	1	1 20	1 45
D-1	1 5	1 10	1 20	2 40	3 30	3 25	15	25	40	50	1 5	1 35
D-2	2	1 55	2 10	2 35	3 30	3 30	30	45	55	1 20	1 25	1 50
D-3	3 35	3 30	3 50	5 45	5 50	6 40	1 50	2 10	2 20	2 40	3	3 20
E-1	4	4 55	5	6 35	7 20	8 15	3	3 40	4 25	4	4 55	6 10
E-2	4 20	4 45	4 55	7	7 45	8 15	3	4 5	4 20	5 30	5 20	6 5
E-3	4 50	4 25	5 15	7 50	6 35	9 25	3 55	4 35	5 15	5 40	6 40	7 10
F-1	9 20	9 10	9 35	(a)	(a)	22 15	3	3 45	4 55	4 25	5 30	8 45
F-2	9 30	9 50	(b)	21 50	22 55	24 15	3 25	3 25	4 10	4 40	6 20	8 40
F-3	9 35	9 45	(b)	23 50	22 45	29 30	3 15	3 55	4 35	4 40	6 35	9

(a) Less than 22 hrs.
(b) More than 10 hrs.

*A, B, C refer to composition as given under "Mixtures" on page 575.

As may be seen by an examination of table V, a very distinct relation exists between the temperature at which the magnesite was calcined and the setting time, that is, increases in the burning temperature resulted in slower setting. Also there is a tendency for the setting time to become slower with a decrease in the percentage of magnesia contained in the mixture, which may be due to a change in both the ratio of magnesium chloride to magnesia and the ratio of the amount of solution to magnesia. Again, there was a tendency for increased fineness of the raw material to result in slower setting, which no doubt may be explained from the fact that at any particular temperature the finer raw material is more readily calcined than the coarser material.

Soundness

The pats used in the setting time tests were employed in comparing different methods for making soundness tests. One pat from each mix was placed in the air of the laboratory after being stored in the moist closet for 24 hours, a second was placed in water and a third was subjected to steam at atmospheric pressure for 5 hours.

None of the air pats up to 28 days showed signs of unsoundness with the exception of those made from mixes B-1AM,[1] C-1AM, C-1BM, C-2AM, and C-2BM which developed radial cracks. Cracks were also observed in the strength specimens made from these mixes appearing sooner in the 2″ x 4″ cylinders than in the briquettes. All the water pats of this series were softened a great deal within 7 days. Some of them were barely brittle with little swelling while others were very soft, swelled or badly cracked. There was very little difference in results produced in the flooring pats as all could be broken easily with the fingers. The mortar pats showed a greater variation in the effects of the water storage but little of definite value was obtained from these tests. Differences occurred more noticeably among the mixes of different proportions rather than among those containing cements burned at different temperatures. The mixes containing the smallest amounts of cement seemed to be less affected than the others, but this was not true in all cases.

[1] In this notation "B" refers to temperature of burning, "1" to sizing (see table I), "A" to mix (see composition page 575), and "M" signifies mortar.

TABLE VI—RESULTS OF STRENGTH TESTS

Tensile Strength—Lbs. per Sq. In.

	Flooring Mixtures, Age									Mortar Mixtures, Age								
	24 hrs.			7 days			28 days			24 hrs.			7 days			28 days		
Burn	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
B-1	287	248	250	283	323	283	528	535	305	247	168	220	270	225	228	385	332	280
B-2	263	260	218	323	293	297	257	215	233	270	212	178	212	185	173	185	130	75
B-3	458	586	545	498	605	653	675	705	760	323	318	238	435	367	347	550	437	322
C-1	337	367	332	360	352	353	287	337	398	358	378	280	283	245	297	193	185	287
C-2	327	338	320	417	372	328	320	380	315	487	402	265	232	208	238	207	248	363
C-3	375	348	367	493	435	440	533	613	628	203	140	298	370	543	407	490	640	703
D-1	543	543	622	620	605	640	515	653	697	362	457	352	530	598	395	452	460	492
D-2	660	575	615	650	630	650	768	723	808	383	380	398	330	558	510	578	572	528
D-3	620	690	590	708	663	645	708	667	762	642	513	382	1,097	893	755	1,113	947	935
E-1	545	515	437	558	658	612	733	748	788	448	442	342	873	692	712	832	825	978
E-2	598	553	470	590	565	760	735	658	878	522	358	295	748	717	637	668	847	797
E-3	573	548	493	862	845	672	882	848	695	445	358	313	757	583	613	758	763	763
F-1	*595	*545	*390	933	862	592	818	867	695	270	237	155	818	718	535	773	642	682
F-2	*557	*420	*300	752	632	482	742	793	692	277	215	110	613	580	470	620	653	603
F-3	*250	*248	*223	373	412	407	650	592	642	212	117	*153	630	518	253	885	793	525

TABLE VI—(Continued)

Compressive Strength—Lbs. per Sq. In.

B-1	490	565	485	590	560	585	2,080	2,025	1,495	1,315	875	855	1,535	1,070	1,125	1,700	1,540	1,910
B-2	620	530	415	540	494	475	480	485	525	1,190	958	692	976	767	692	1,025	845	690
B-3	1,425	1,220	1,240	1,820	1,590	1,745	2,380	2,475	2,335	2,170	1,755	1,625	2,645	2,160	1,750	2,490	2,030	2,140
C-1	665	587	835	515	490	745	420	580	805	1,905	1,735	1,350	1,950	1,570	1,340	1,745	1,535	1,595
C-2	695	655	545	681	674	652	510	750	895	1,565	1,428	1,070	1,075	1,350	1,150	1,340	1,545	1,510
C-3	1,060	865	905	1,085	930	1,060	2,085	1,590	1,775	1,545	1,425	1,535	2,190	1,620	2,005	2,445	2,650	2,785
D-1	1,685	1,835	1,800	2,030	2,230	2,160	2,090	2,785	2,755	1,975	2,650	2,150	2,070	3,075	2,475	2,500	3,320	3,110
D-2	2,765	2,510	2,200	2,905	2,675	2,620	3,510	3,265	3,265	2,630	2,535	3,140	2,870	...	3,645	3,270	...	3,990
D-3	3,260	3,160	2,830	3,900	3,300	3,055	4,710	4,270	4,125	6,480	5,180	3,920	7,910	6,790	5,210	9,540	7,730	7,010
E-1	2,865	2,720	2,140	3,975	3,990	3,525	4,420	5,150	4,920	5,210	4,375	3,045	8,040	6,840	4,870	9,300	7,660	6,400
E-2	1,980	2,070	2,370	3,250	2,775	3,685	3,795	3,315	4,340	5,500	3,170	2,840	7,850	7,020	4,780	8,550	7,930	5,880
E-3	3,170	2,855	2,135	5,165	4,430	4,040	6,820	6,250	5,925	5,980	3,980	2,840	8,810	6,610	4,735	8,770	7,815	5,915
F-1	*2,585	*2,105	*1,525	4,500	3,770	3,240	5,610	5,050	5,010	2,510	2,060	730	7,730	5,585	4,020	8,375	5,060	4,490
F-2	*2,100	*1,585	*1,020	3,925	3,475	2,870	5,780	4,195	4,720	2,450	1,795	600	6,950	4,985	3,695	7,250	5,680	4,990
F-3	*1,095	*835	*650	3,815	2,980	2,500	6,690	6,690	5,510	2,040	950	*942	6,045	4,770	2,815	6,270	6,140	5,680

*Tested at 48 hours on account of slow setting.

The steam test gave results similar to those obtained by storing the pats in water, and the same general remarks as to effects of water storage may be repeated here. The steam test, however, was considerably more severe.

Strength Determination

Strength test specimens were made in both tension and compression. The Standard briquette and method of molding for testing Portland cement were used in the tension tests and 2″ x 4″ cylinders in the compression tests. The specimens were not placed

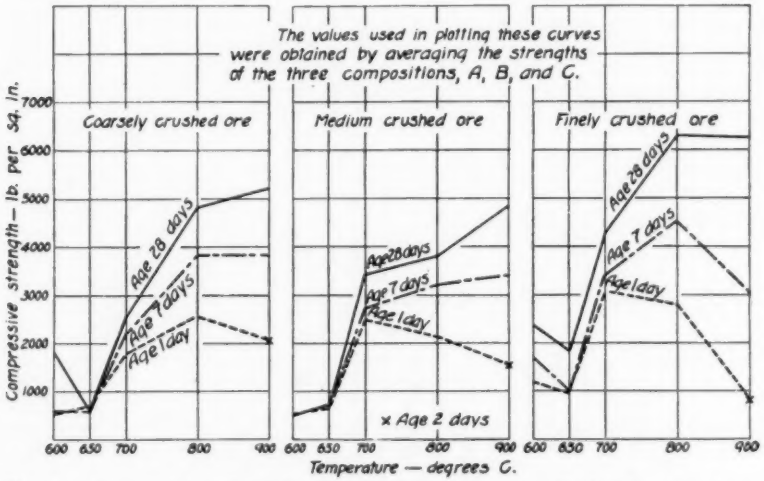


FIG. 1.—Compressive strength of flooring mixtures as affected by size of ore and burning temperature.

in a damp closet after molding but were protected from loss of moisture to the atmosphere by covering tightly with glass plates. At the end of 24 hours, they were removed from the molds and stored in the laboratory freely exposed to the atmosphere until tested.

The results of the strength tests are given in table VI, each being an average of 3 tests. The effects of the calcining temperature and size of ore on the strength of the flooring and mortar mixtures are shown in figures 1 and 2, respectively. In order to avoid the use of the large number of curves necessary to show the tests in detail the results obtained from averaging the strength

of the three mixes of different proportions (A, B, and C) containing magnesia from the same burn were plotted.

These data show that there is an optimum temperature required for burning the magnesite, under the conditions used in these tests, to develop the best strength at the early period. Apparently the magnesia burned at 600 and 650°C was underburned, though the question arises as to whether or not the magnesia was more active and required more chloride than actually

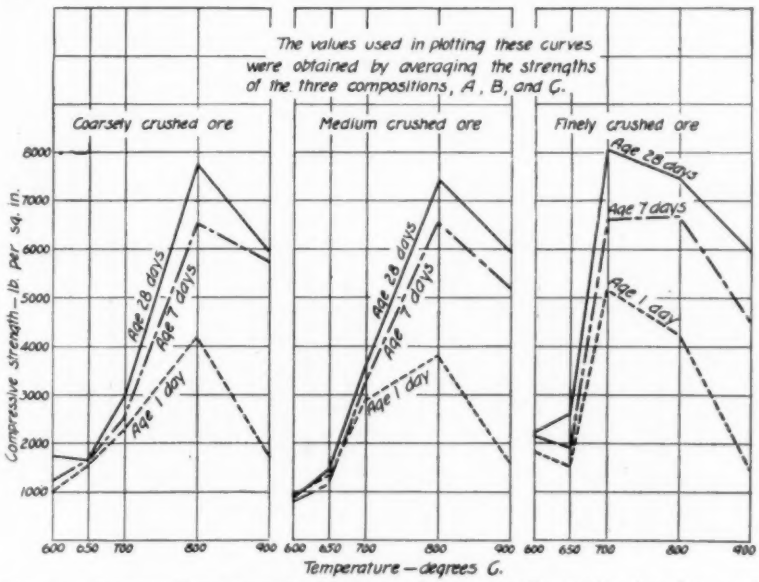


FIG. 2.—Compressive strength of mortar mixtures as affected by size of ore and burning temperature.

used. The disintegration of the test piece made from such burned material at late periods of storage and the results given in table VIII would indicate that under-burning was not the reason for the low strength of the low burned magnesia, but rather the use of too small an amount of chloride. Over-burning is apparent in the results obtained from the test pieces made from the finely crushed magnesia burned at 800 and 900°C. But this is manifested only by low early strength, as at the late periods the strength furnishes few data to assist at arriving at a proper burning temperature.

Comparing the strengths developed by the calcining of the coarse, medium, and fine ground ore at any one temperature it is clearly evident from the 24-hour strength alone that the fine ground material is the more readily burned. Hence after low burning it is the more active, while after calcination at the higher temperatures it is the more inactive.

Figures 3 and 4 are for the purpose of comparing the 28-day strengths obtained in the use of different proportions of magnesia and aggregate. There is very little difference in the strengths of the three flooring compositions, and even among the mortars,

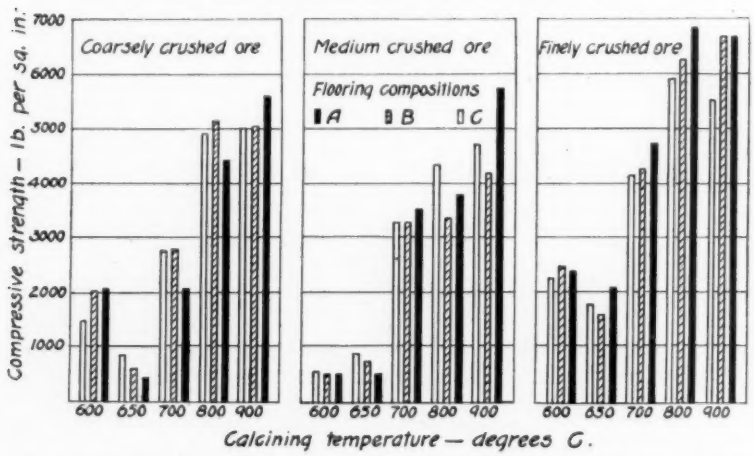


FIG. 3.—Comparison of the compressive strengths of the various flooring compositions at the age of 28 days.

in which the percentage of magnesia varies from 11 to 33 per cent, there is generally comparatively little difference in most cases. The maximum increase in strength resulting from a 200 per cent increase in magnesia content was only 87 per cent. This difference of strength between the lean and rich mixtures occurred with the cements from the burns at the higher temperatures which were presumably the less active. These facts again suggest that low strength was not an inherent quality of the low burned magnesite, as in that case an increase in the proportion of magnesite within limits should have resulted in higher strengths. In other words,

the low strength may have been due to the increased activity of the magnesia, advantage of which was lost by the use of the same amount of chloride as used in the more inactive product.

Another interesting relation derived from the strength tests is shown in figure 5. It was noticed that the ratio of tensile strength to compressive strength varied a great deal in the tests on the

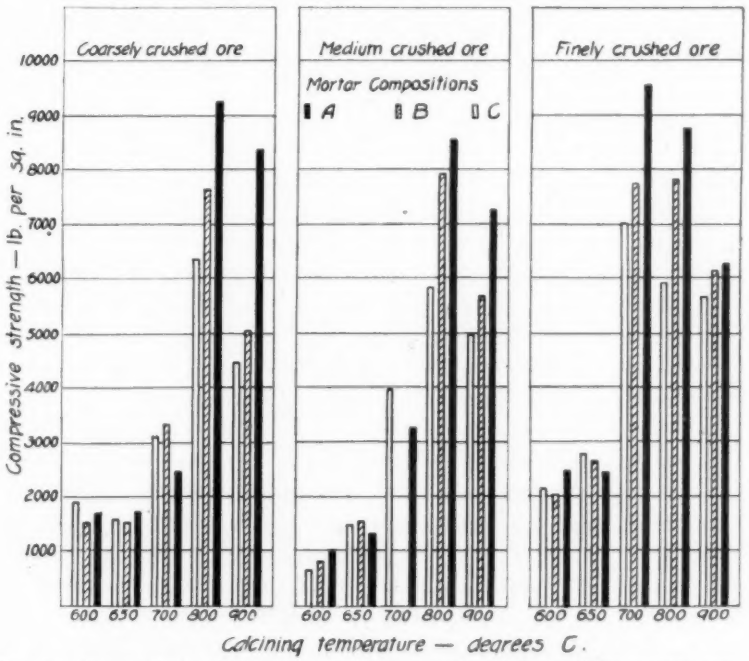


FIG. 4.—Comparison of the compressive strengths of the various mortar compositions at the age of 28 days.

flooring mixtures. By plotting the compressive strength against the ratio of tensile to compressive strength, it was found that all points fell in the close vicinity of a smooth curve, regardless of the magnesia or the proportions used. It may be seen that the tensile strength varies from approximately 10 per cent to 80 per cent of the compressive strength and that the ratio increases as the strength decreases. All results of the 28-day strength tests of the various flooring mixtures were used. The point to be emphasized is that

the relation between tensile and compressive strength is not a straight line relation in this type of mixtures, as is often assumed, but that the ratio of tensile to compressive strength varies considerably with the strength.

The variation in this ratio is a great deal less among the mortar tests, being from about 8 per cent to 24 per cent. Furthermore, the relation between tensile and compressive strengths, although somewhat similar to the above, is much less obvious.

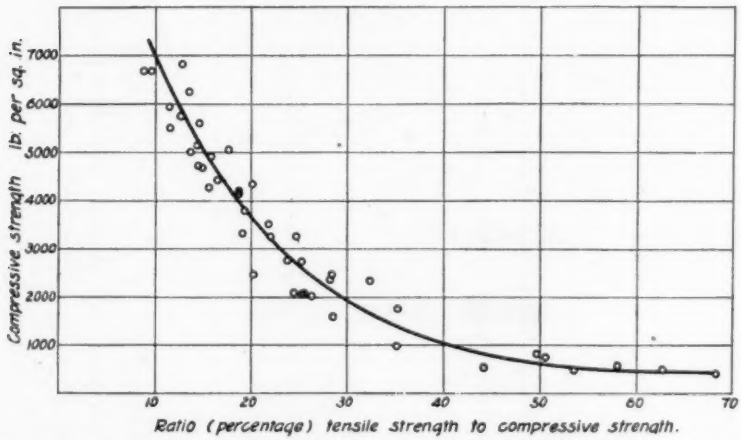


FIG. 5.—Relation of tensile to compressive strength of the flooring mixtures at 28 days.

Change in Volume

The linear changes of specimens 1 inch by 1 inch by 14 inches long were measured by means of a Berry strain gauge. These bars were molded in the same manner as the briquettes, but the upper surface was left exposed to the air during the first 24 hours. They were then removed from the molds and stored in the air of the laboratory in a horizontal position supported at two points one-fourth the length from the ends with all sides exposed.

Warping to a greater or lesser degree occurred in nearly all bars, usually beginning before the 28-day period. In nearly every case, the warping has been in the vertical plane with respect to the position in which it was molded and stored, with a bowing up of

the centers of the bars. It was thought that the manner in which they were stored might influence the warping, so one bar of some of the sets was stored with the side molded last facing down. These bars warped with the centers bowing down which showed that the warping was influenced to little extent by the storage position. The reason for this warping or unequal change in lengths of the top and bottom of the specimen is not evident. Without doubt errors were introduced in the results of these measurements on account of this, but since the change in volume was usually comparatively large in cases of bad warping, very accurate results were unnecessary.

The results of these tests expressed in thousands of a per cent are given in table VII. The initial readings in a large number of cases were made at the time of final set but these are not shown. To make the table consistent throughout all calculations were based on the 24-hour reading. It may be seen that the behaviors of the various mixtures appear very erratic and very little relation seems to exist between change in volume and any of the other properties of the mixes or conditions of tests. This was somewhat disappointing in view of the fact that a greater knowledge of the causes and of means for preventing excessive changes in volume was most desired. At least a test by which materials undesirable in this respect may be excluded is essential before magnesia compositions can be classed as a reliable building material. No very general conclusion can be drawn from the data. They indicate only certain trends—as the rich mixes expanding more than the lean ones the test pieces for the high burned magnesia expanding more than that from the low burned, and the more general constancy of the medium burned product.

Miscellaneous Tests

As previously mentioned, specimens from a number of mixes containing magnesia calcined below 700° showed signs of disintegration within a comparatively short time. The only plausible explanation of this seemed to be that there was an unusual amount of highly active magnesia present owing to the conditions of calcining and that the amount of magnesium chloride was insufficient to complete the reaction in the formation of magnesium

TABLE VII—RESULTS OF CHANGE IN VOLUME TESTS

Flooring Mixtures

Linear Changes in Thousandths of a Per Cent—between the Ages of 24 Hrs. and

	48 hrs.			4 days			7 days			28 days			3 mos.		
Burn	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
B-1	+319	—36	—13	+3	—88	—24	+191	—84	—41	+14	—92	—22	+28	+50	+282
B-2	—25	—26	—25	—25	—38	—46	—27	—46	—67	—(a)	—(a)	—(a)	—(a)	—(a)	—(a)
B-3	+24	+18	—3	+3	+17	—9	—25	—6	—1	—143	—72	0	—133	—51	+4
C-1	+45	+22	+6	+57	+27	—1	+86	+45	+8	+253	+118	+49	+487	+221	+109
C-2	+27	+8	+4	+93	+51	+29	+200	+105	+52	+217	+104	+112	+557	+292	+170
C-3	+14	+17	+13	+9	+11	+15	+22	+4	+25	+10	+18	+26	—33	—26	+26
D-1	+18	+15	+26	+40	+20	+12	+32	—6	+5	+27	+21	+15	—75	—122	—14
D-2	+48	—104	+48	+76	+61	+47	+77	+75	+48	+70	+64	+46	+4	—11	+49
D-3	+98	+118	+114	+142	+129	+137	+111	+107	+127	+82	+90	+126	+123	+111	+130
E-1	+174	+114	+140	+228	+155	+193	+218	+154	+187	+225	+153	+190	+209	+140	+172
E-2	+411	+176	—103	+483	+239	—57	+494	+247	—47	+489	+244	—52	+481	+235	—59
E-3	+153	+135	+78	+227	+202	+126	+211	+191	+130	+224	+199	+132	+227	+200	+130
F-1	+161	+167	+150	+347	+373	+360	+421	+504	+558	+406	+492	+564	+395	+483	+548
F-2	+180	+167	+128	+437	+428	+286	+(a)	+(a)	+488	+(a)	+(a)	+(a)	+(a)	+(a)	+(a)
F-3	+87	+71	+41	+45	+201	+140	+327	+464	+378	+360	+551	+507	+347	+513	+489

TABLE VII—(Continued)

Mortar Mixtures

B-1	—11	+5	—10	—9	+16	—14	—52	—16	—45	+53	—75	+17	+17	—68	+30
B-2	+77	+27	—11	+77	+49	—16	+432	+72	—21	+(a)	+199	—254	+(a)	+118	+357
B-3	+15	+19	+14	+11	+22	+23	—8	+7	+11	—22	+34	—15	—8	+294	—17
C-1	+9	+73	+5	+232	+163	+7	+576	+325	+17	+(a)	+(a)	+63	+(a)	+(a)	+138
C-2	+79	+71	+4	+408	+390	+26	+(a)	+(a)	—33	+(a)	+(a)	+77	+(a)	+(a)	+243
C-3	...	+20	+12	...	+43	+12	..	+95	+8	...	+139	+1	...	+463	+12
D-1	+3	+1	+7	+38	+30	+16	+73	+49	+9	+131	+137	+11	+(a)	+391	—20
D-2	+4	+12	+11	+39	+34	+18	+66	+49	+16	+131	+98	+27	+(a)	+303	+19
D-3	—8	+9	+30	—28	+7	+37	—31	—10	+31	—9	—7	+22	+54	+24	+37
E-1	+11	+29	+79	—3	+30	+100	+5	+27	+105	+22	+36	+103	+104	+73	+99
E-2	+15	+59	+101	0	+53	+132	+9	+58	+138	+23	+70	+141	+95	+100	+134
E-3	+41	+78	+61	+35	+84	+84	+48	+92	+85	+64	+98	+86	+162	+134	+85
F-1	+197	+178	+141	+284	+235	+220	+253	+268	+340	+347	+269	+349	+337	+244	+338
F-2	+190	+176	—141	+254	+274	—38	+312	+315	+110	+367	+319	+118	+359	+300	+71
F-3	+208	+135	+5	+353	+322	+71	+384	+401	+123	+434	+401	+161	+410	+396	+152

+(a) Expansion beyond limits of instrument.
—(a) Contraction " " " "

oxychloride. As a consequence the remaining active magnesia slowly hydrated with the accompanying increase in volume and disintegration of the test specimen. In accordance with this reasoning, magnesite cement mixtures containing an insufficient amount of magnesium chloride might disintegrate in time, depending upon conditions such as amount of excess active magnesia present, moisture conditions, type of aggregate and the proportion of magnesia to aggregate and to chloride. Also, the rate at which such hydration takes place would depend largely upon the extent to which the magnesite was burned.

For the purpose of obtaining further information in this direction, the mixture which disintegrated to the greatest extent was duplicated and gauged with solutions of higher concentrations. The test specimens which were gaged with the same strengt solution (22°Bé.) as was previously used failed to behave as expected in that no excessive expansion or disintegration occurred within the same period, although there was a retrogression in strength with age. This would indicate that the original magnesia had undergone some change during storage. No doubt, owing to its activeness toward moisture, partial hydration had taken place, making the ratio of magnesium chloride to active magnesia higher in the later tests. However very marked increases in strength resulted from the use of the higher gravity magnesium chloride solutions, and this prompted the following series of tests.

Magnesite crushed to the fine size and calcined at 650° was used in the various compositions previously described and each of these was gaged with several solutions of different gravities.

The results of the strength tests are given in the first part of table VIII. It may be seen that by means of more concentrated solutions the compressive strength of each of these mixes was increased approximately from 300 to 600 per cent over that resulting from the use of the 22°Bé. solution. It seems therefore, that very much greater economy in the use of magnesite cement may be possible, that is, by the use of somewhat more concentrated solutions, a specified strength may be attained with considerably smaller proportions of magnesia. Furthermore, maximum strength does not result from any definite ratio between the magnesium

TABLE VIII—STRENGTH OF COMPOSITIONS AS AFFECTED BY CONCENTRATION OF SOLUTION AND CONSISTENCY

Ratio

TABLE VIII—STRENGTH OF COMPOSITIONS AS AFFECTED BY CONCENTRATION OF SOLUTION AND CONSISTENCY

Composition	Marked	% Sol.	Ratio $MgCl_2$ / MgO	Sol[1], °Bé.	Strength (lbs. per sq. in.) Tensile 1 day	Tensile 7 days	Tensile 28 days	Compressive 1 day	Compressive 7 days	Compressive 28 days
AF	4–1	72	.290	22	375	493	533	1060	1085	2085
	4–2	72	.333	25	592	657	760	1235	1605	2775
	4–6	72	.468	34	902	965	907	6325	7305	7950
BF	4–3	68	.322	22	348	435	613	865	930	1590
	4–7	68	.520	34	1008	932	958	4840	5395	8060
CF	4–4	67	.385	22	367	440	628	905	1060	1775
	4–5	67	.500	28	758	742	790	3560	3760	4475
	4–8	67	.622	34	638	883	907	3500	4575	5980
AM	4–9	27	.169	22	203	370	490	1545	2190	2445
	4–12	27	.188	25	205	437	470	2115	3495	4285
	4–15	27	.239	31	543	985	880	7570	8820	9580
	4–17	27	.266	34	700	713	238	7405	8780	*8460
BM	4–10	22	.201	22	140	543	640	1425	1620	2650
	4–13	22	.261	28	415	780	415	5290	7080	6180
	4–18	22	.325	34	793	1173	1042	9410	8910	10950
CM	4–11	18	.329	22	298	407	703	1535	2005	2785
	4–14	18	.427	28	510	607	878	5315	6210	7120
	4–16	18	.478	31	477	540	770	5240	6000	6600
	4–19	18	.532	34	542	615	570	5175	5190	8800
CF	5–5	56.9	.327	22	433	535	628	945	1245	2340
	5–6	76.6	.440	22	455	483	617	975	1075	2535
	5–7	87.0	.500	22	370	445	605	800	1034	2105
	5–8	108.3	.622	22	283	347	528	620	854	1795
CM	5–1	15.2	.278	22	237	427	547	1690	2070	3045
	5–2	20.5	.375	22	275	397	625	1700	2170	3890
	5–3	23.3	.427	22	298	377	552	1800	2090	3900
	5–4	26.1	.478	22	258	348	547	1705	2020	3485

*Spalled badly before failing.

chloride and magnesia. This statement is conclusive from the above data, together with the results of tests given in the second part of table VIII. In the latter series the ratio of magnesium chloride to magnesia was varied in each mixture by changing the percentage of solution rather than by changing the strength of solution. Under these conditions the strength was not increased by an increase in the above ratio as in the former series, which shows that the consistency (and perhaps the ratio of solution to magnesia) is an important factor.

There are so many interesting and vital facts that can be brought out from the data in this table that it seems well to call particular attention to them. Thus the relation between the amounts of magnesia in the different mixes should be recalled,—the AF mix contains 50 per cent magnesia, BF 42.5 per cent and CF 35 per cent. However note that the latter when gaged with 28° Bé. solution gives as good tensile strength as the AF mix when gauged with 25°Bé. solution and far better compressive strength. In the mortar the AM mix contains 33 per cent magnesia, the BM mix 22 per cent and the CN mix 11 per cent. Here again it is evident that the latter mix with a chloride solution of greater concentration (28°Bé.) than commonly used produces practically as good tensile and compressive strength, especially at late periods, as the rich AM mix with a solution of either high or low concentration. However too sweeping deductions can not be made from such comparison of the mixes, as a close study of the data shows that each mix requires a study of the amount and concentration of the chloride used to produce the best strength. If it were a matter alone of chloride-oxide ratio, then the different flooring mixes in which such ratios as .333 and .322 or .500 and .520, and the mortar mixes in which ratios as .188 and .201 or .266 and .261 or .325 and .329 were used should give somewhat comparable results, but such is not the case. This is due almost undoubtedly to the fact that a differently proportioned mix of the same dry constituents was used. Hence as stated above the problem is not only one of consistency (amount of solution used) or chloride-oxide ratio but also the proper proportioning of the dry mix.

The striking feature of the data however is the fact that with such a very active magnesia as that used in this part of the investigation it is not possible to develop its qualities by the use of a wet consistency (or high percentage) of 22° Bé. solution. In the mortar mixes, 4–14 is comparable with 5–3, in that they are identical dry mixes, but the former was gauged with 18 per cent of 28°Bé. solution and the latter with 23.3 per cent of 22°Bé. solution, producing in both cases the same chloride-oxide ratio. But the use of the higher gravity solution produced by far the better strength. The same relation exists between the dry mixes of 4–16 and 5–4 and when the consistency of the two was changed, though the chloride-oxide ratio was maintained the same by the use of solutions of different gravity, better results were produced by the use of the more concentrated solution. Similar relations exist in the flooring mixes and hence similar comparisons can be made between mixes 4–5 and 5–7, or 4–8 and 5–8 and again it will be noted that the increased strength has been produced by the greater concentration of the solution. Or the matter may be stated in another way by saying that a larger amount of a relatively weak solution (22°Bé.) will not give with any one mix as high strength as a less amount of a strong solution.

Before leaving the study of these data it would be well to call particular attention to some of the very high strengths that have been obtained, especially with the very lean mixes, as for instance the flooring mix CF (758 and 638 lbs. per sq. in. in tension and 3560 and 3500 lbs. in compression at the end of 24 hours) when either the 28° or the 34° Bé. solution, respectively, was used. The flooring mix 4–7 containing 7.5 per cent more magnesia developed greater strength, but the strength of the 4–8 mix is more than that required for practically all purposes and the more economical. The mortar mixes containing but 11 per cent magnesia also developed very high strength with the more concentrated solutions, these ranging from 477 to 542 lbs. per sq. in. in tension at 24 hours and 570 to 878 lbs. at the end of 28 days; and in compression ranging from 5175 to 5315 lbs. per sq. in. at 24 hours and 6600 to 8800 lbs. at the end of 28 days. The highest values of any mix, either flooring or mortar, were 1173 lbs. per sq. in. in tension at the

end of 7 days for the mortar mix containing 22% magnesia, gaged with 34° Bé. solutions, and 10950 lbs. per sq. in. in compression at the end of 28 days for the same mix similarly gaged.

The manner in which setting time was affected is shown in table IX. In practically all cases the time of setting of a mixture was lengthened by an increase in the concentration of the solution.

TABLE IX—RESULTS OF SETTING TIME TESTS

Composition	Marked	Per cent solution	Ratio $\frac{MgCl_2}{MgO}$	solution °Bé'.	Initial hr.	Initial min.	Final hr.	Final min.
AF	4–1	72	.290	22		20	1	50
	4–2	72	.333	25		25	1	50
	4–6	72	.468	34	1	25	2	40
BF	4–3	68	.322	22		30	2	50
	4–7	68	.520	34	1	25	2	50
CF	4–4	67	.385	22		30	2	45
	4–5	67	.500	28		45	1	60
	4–8	67	.622	34	1	45	3	30
AM	4–9	27	.169	22		22		10
	4–12	27	.188	25		5		10
	4–15	27	.239	31		10		35
	4–17	27	.266	34		20	1	45
BM	4–10	22	.201	22		4		15
	4–13	22	.261	28		10		25
	4–18	22	.325	34		35	1	40
CM	4–11	18	.329	22		10		30
	4–14	18	.427	28		20	1	
	4–16	18	.478	31		40	1	10
	4–19	18	.532	34	1	15	2	45
CF	5–5	56.9	.327	22		20	2	20
	5–6	76.6	.440	22		45	2	55
	5–7	87.0	.500	22	1	25	4	20
	5–8	108.3	.622	22	2	10	5	10
CM	5–1	15.2	.278	22		10		20
	5–2	20.5	.375	22		15	1	15
	5–3	23.3	.427	22		25	1	45
	5–4	26.1	.478	22		35	1	50

This is contradictory to the results obtained by some investigators. It was thought possible that the difference was due to the use of fresh low burned magnesite in one case and aged or higher burned magnesite in the other. Accordingly tests were made on magnesia from the F-3 burn and on two commercial compositions. The results of the tests on the high burned magnesia were similar to the above but even more pronounced, whereas the commercial mixtures set somewhat more quickly as the concentration of the solution was increased. Other tests of this nature lead to the conclusion that the apparently conflicting results were probably normal, which may be explained by the fact that sufficient exposure of magnesia to allow considerable hydration to take place will reverse the effects of increasing the concentration of the solution as compared with the effects on freshly calcined material.

Another point of importance is that higher concentrations of the solutions resulted in greater resistance to the action of water or steam on the set material. This was true without exception. The pats of all mixes gaged with a 22°Bé. solution became soft in the steam test but marked improvements were observed with each increase in the concentration of the solution. In fact the mixes containing the smallest proportion of magnesia and gaged with the 34° Bé[1]. solution appeared to be little affected by the steam treatment. It is therefore probable that greater resistance of stucco to the weather may be accomplished in this way.

However, before the use of strong solutions can be judiciously recommended, more comprehensive and conclusive data pertaining to the effects on volume changes is necessary. The results of expansion tests on the mixes of series 4 up to the 28-day period show that during this time the change in volume is influenced considerably, in some instances increased and in others decreased. The concentrated solutions caused the flooring mixtures to expand during the first 48 hours, but at the 28-day period the volume was approximately the same as the original, while those gaged with the 22°Bé. solution showed only a slight tendency toward expansion at the earlier periods and considerable contraction at 28 days. In contrast with the above, increased concentrations of the solution caused very great contraction of the richest mortar mixtures during the first 24 hours.

Another dissimilar behavior of this same magnesia occurred in the tests in which only the amount of solution was varied (series 5). In this instance only the flooring mixtures were affected to any considerable extent. The driest flooring mixture contracted 0.2 per cent in 28 days and as the percentage of solution (or the ratio of chloride to oxide) was increased, the contraction decreased until finally the highest percentage of solution resulted in an expansion of approximately 0.05 per cent. This further demonstrates the fact that a satisfactory explanation of the cause of changes in volume of such compositions is far from simple. There is no doubt that an explanation would involve a number of factors, some of which are dependent on others for their effectiveness in producing changes in volume. It would seem to follow, therefore, that such material as produced and used in practice, with the added variables necessarily introduced, presents a still more difficult problem in the matter of developing methods for avoiding the excessive changes in volume which persistently occur from time to time. Fortunately this does not seem to be the case. Limited tests indicate that in most of the stuccos and floorings the stresses developed due to a tendency toward a change in volume in the direction which might cause large cracks or buckling are usually sufficiently resisted by the high strength of the material itself, by its strong bonding power, and by the reinforcement, such as the fibrous aggregate, metal, or the material acting as the base. It may be that only the mixtures showing a very decided change in volume in the laboratory tests as made would give trouble in practice, providing, of course, that the material were properly placed. If this is true, then the problem is much less complicated than the laboratory tests might lead one to believe.

DESIGN OF A FURNACE FOR ANNEALING OPTICAL GLASS[1]

By Donald E. Sharp

ABSTRACT

Design of a natural-gas furnace for annealing optical glass.—Most of the furnaces built for annealing regular glassware are unsuitable for optical glass due to irregularity and inequality of temperatures. *Working drawings* are given for a successful *optical glass annealing furnace* operated by *natural gas*. The design is novel in the placing of the flues and burners in such a manner as to supply the heat and remove it in a symmetrical manner, thus obtaining uniformity of temperature.

In the course of the manufacture of optical glass in this country during the early days of the war, little was known of the design and construction of the furnaces which had been employed for this work abroad, and it was consequently necessary to rely on the experience of glass furnace builders for designs. Suitable designs for melting furnaces presented little difficulty to the few contractors whose services were available, but the annealing furnaces built were in some cases quite unsuitable for the careful treatment necessary in the proper annealing of optical glass. There were, to be sure, many lehrs and ovens already in successful operation in the glass factories of the country, but the heat treatment necessary for the successful annealing of bottles and thin articles of glass was found to be a much simpler process than the manufacture of large glass slabs and prisms of optical glass, which were sufficiently free from strain to enable their use in optical instruments of precision. Although the annealing temperatures of the glasses, and the proper cooling rates, had been determined, it was in many cases a hopeless task to attempt to hold the proper temperature for the length of time required, or to cool at the correct rate with the furnaces which were available.

The furnaces which were first used by the author in the early days of the war, were of the oven or kiln type, and were designed

[1] Received March 7, 1921.

and built by a contractor who was experienced in the manufacture of such furnaces for the bottle trade. The annealing chamber proper was approximately 4 ft. wide by 6 ft. long, the arched crown being about 18″ from the floor of the chamber and having a spring of about four inches. These furnaces would undoubtedly have been perfectly satisfactory for annealing bottles or tableware, but were entirely unsuccessful in annealing slabs of optical glass. The chief causes of their failure were combustion faults arising from poor burner and combustion chamber design. The temperature distribution was decidedly poor, and the free cooling rate so great that considerable fuel was necessary to maintain the furnace at the annealing temperature.

The fuel available was natural gas of 1120 b. t. u. per cubic foot supplied at four ounces pressure, but this fuel, though an excellent one, required a great quantity of air for perfect combustion. The burners used were eight in number, four on each side. Each burner consisted of a $1^1/_2$″ pipe extending through the side wall to the crown of the furnace and fitted on the outside with the ordinary commercial bunsen type of air-gas mixer. The jet opening was poorly proportioned to the gas pressure, the size of pipe, and the natural draft of the furnace, and the result was a yellow flame, quite prone to "strike back," and heating a more or less local area. No trouble with the burners was encountered while the door of the furnace was open and the furnace being filled with glass, but it was frequently found that soon after closing the door the oxygen in the chamber had been entirely consumed, and the flame extinguished because the mixers were not functioning properly.

The draft flues were in the conventional position, in the back wall of the furnace. The back was consequently always hotter than the front of the chamber, and the effect of this hotter portion combined with the effect of the local hot areas from the individual burners was to cause inequalities in temperature amounting to from 50° to 100°C.

The rapid free cooling rate of the furnace was due to the use of only eight inches of red brick as an insulating medium. The outer walls of the furnace and the crown were so hot when the

furnace was in operation that they could not be touched. The expense of fuel used to supply heat lost by radiation was consequently considerable.

In the absence of any better designs or any precedent by which to be guided, the author was prompted to take this work in hand, and as a result produced a rather unique design which not only overcame all of the faults of the furnace mentioned, but was found to require much less attention from the furnace operator. The worst of the faults were removed simultaneously by altering the construction and type of the burners, their position, and the position of the flues.

The burners used in the new design were of the perforated tube type; the size of jet and diameter of the burner holes which gave the best results being determined by trial. These dimensions and the general construction are shown in figure 1. It was found desirable to have many small holes rather than a few large ones in order to prevent a tendency to "strike back" when operat-

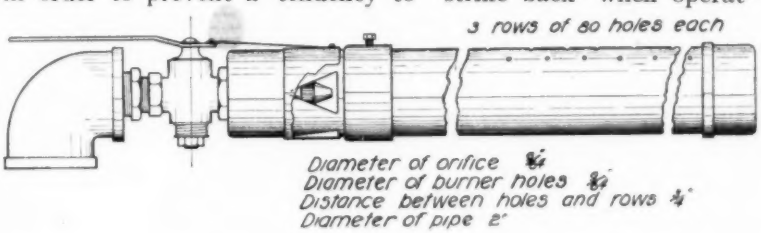


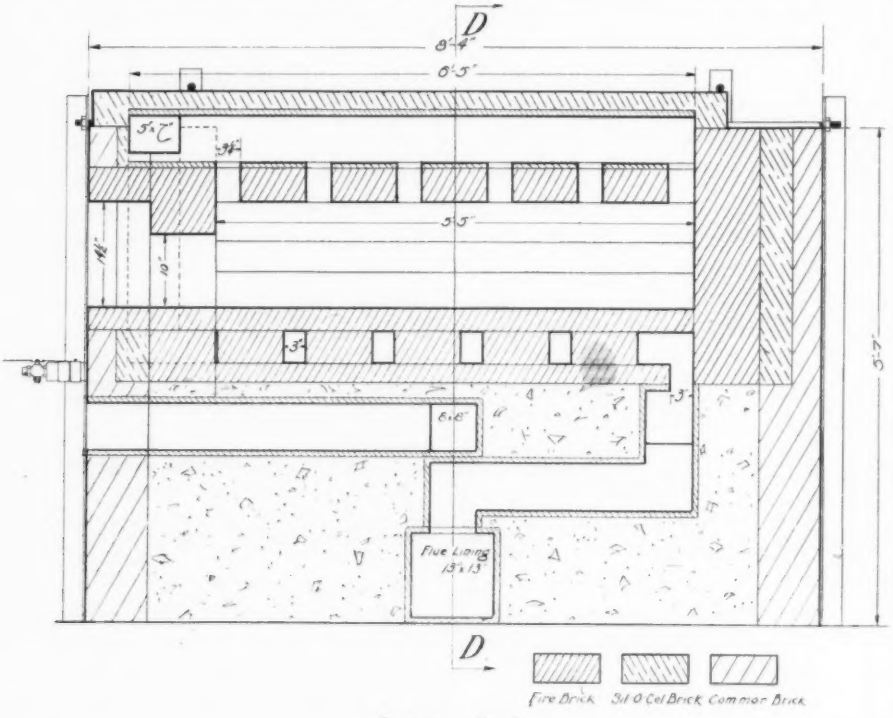
FIG. 1.

ing at a low temperature. The total area of the holes was made sufficiently great so that there was no back pressure or chance for a smaller volume of gas-air mixture to issue through the holes than the volume required by the dimensions of the mixer jet.

The system of flues, which may be understood readily by referring to the figures, is in the opinion of the author an original one. The auxiliary air ducts below the burner pipe insure a large enough supply of air to give perfect combustion even when the furnace is running at a high temperature, and prevent the flame from rising above the pipe as frequently happens when a large volume of gas is fed to a bunsen burner in a closed chamber.

The hot products of combustion pass up and over the breast wall to the furnace chamber and thence through holes in the top of the crown. It should be noted that the heat is thus supplied to the furnace in an entirely symmetrical manner.

The flue on top of the furnace conducts the products of combustion to two vertical flues in the front of the furnace, from which

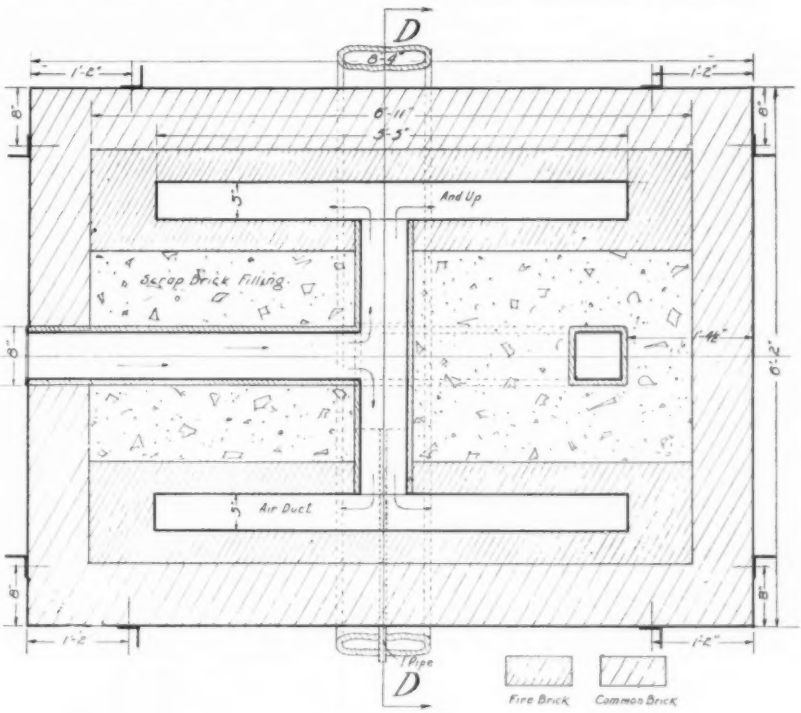


Section A-A

they pass into the large chamber directly under the furnace floor. This arrangement of the flues permits a greater transfer of heat to the front of the furnace which is normally the coldest portion, and removes a source of higher temperature in the already too hot rear of the furnace. The heat produced is, therefore, made almost completely to encircle the glass in the chamber, the rear alone being neglected in this respect. By actual test the tem-

perature was found to vary by not more than five degrees centigrade throughout most of the chamber. Near the door the temperature was slightly lower than in the chamber proper, as was to be expected.

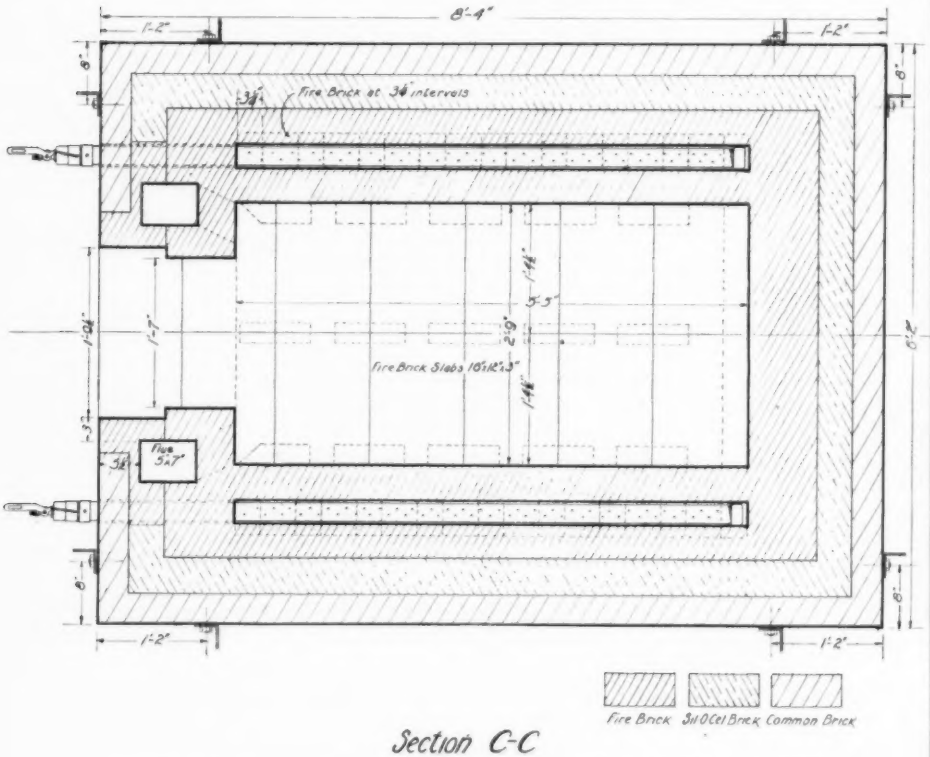
The mechanical construction of the furnace is easily ascertained from the figures. For the eight-inch red brick wall used



Section B-B

in the old furnaces, there was substituted four inches of red brick and four and one-half inches of Sil-O-Cel insulating brick. This additional insulation aided materially in reducing the loss by radiation and therefore the free cooling rate. At first this insulating wall was planned to be nine inches thick, but the thinner wall was decided on by reason of the necessity of building the new furnaces on the foundations of the old ones.

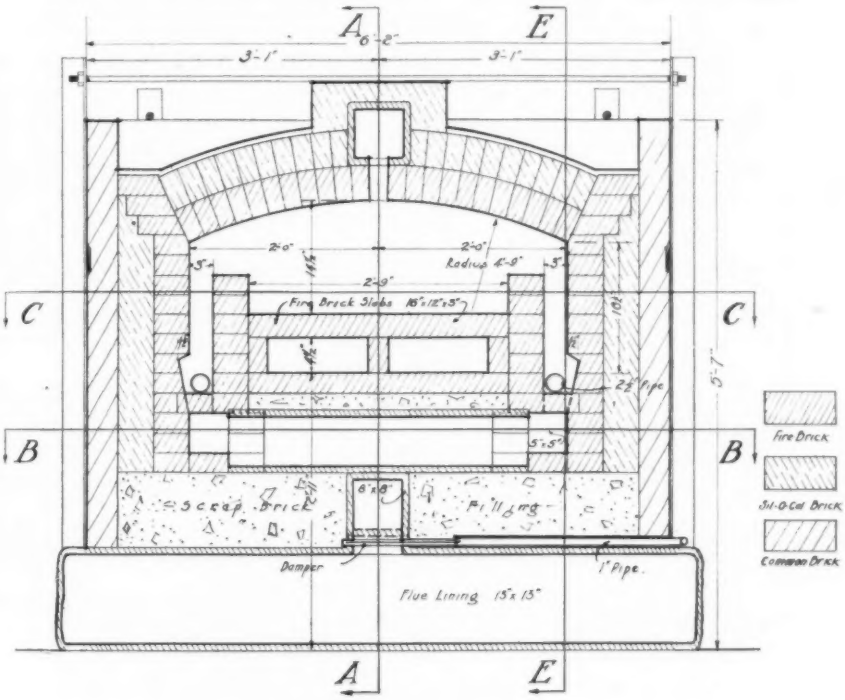
The operation of the furnace was very simple. The stack draft was opened, the gas turned on and lighted and the auxiliary air vent closed; as the temperature of the furnace increased the air vent was opened a little at a time and only enough to prevent the flame from becoming luminous. Any greater amount of air admitted would merely mean that the flame would be



Section C-C

cooled by the excess air. Peep holes which were cut through the front of the furnace and just above the burners, made it easy to ascertain the character of the flame when necessary. The furnace temperature having increased to the proper degree, the gas valve was set at the proper position to hold the temperature; this position being known by trial, and the air vent closed as far as consistent with proper flame conditions.

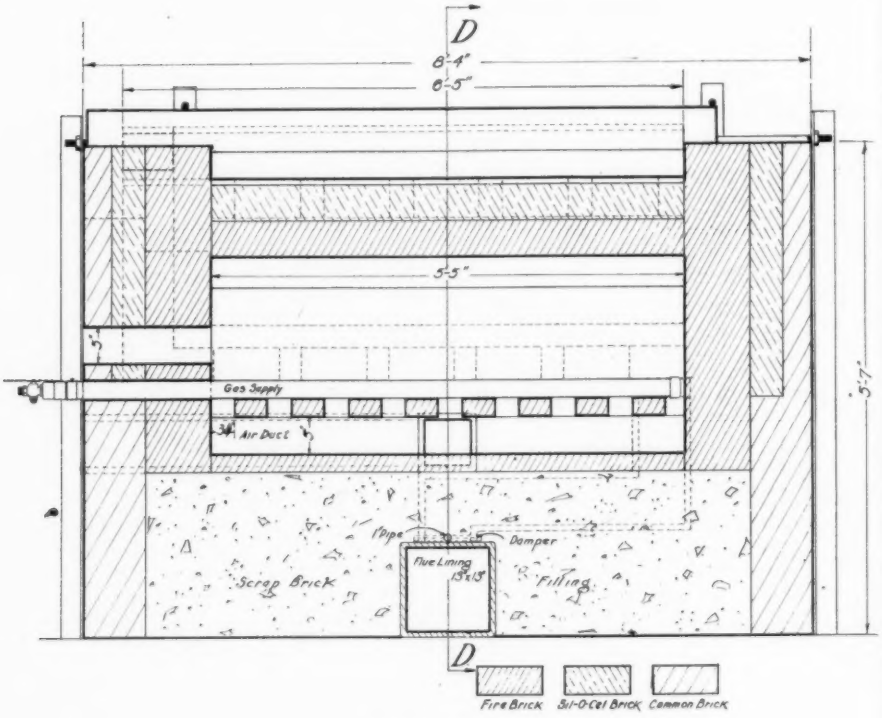
The work was then filled into the furnace, which took an average of nine hours time, and the annealing process started. If the schedule called for holding the furnace at a definite temperature this was accomplished with little attention from the operator, but the slow cooling over the first drop of 50 to 100 degrees necessitated more careful attention. The regulation



Section D-D

was accomplished by the manipulation of the main gate valve according to the personal judgment of the operator.

After the furnace had cooled to the point where its free cooling rate was so low as to prevent permanent strain, the gas was shut off and the dampers, peep holes and so forth, closed and sealed. After a drop of about a hundred degrees further, the stack dampers and air vent were opened slightly. From time to



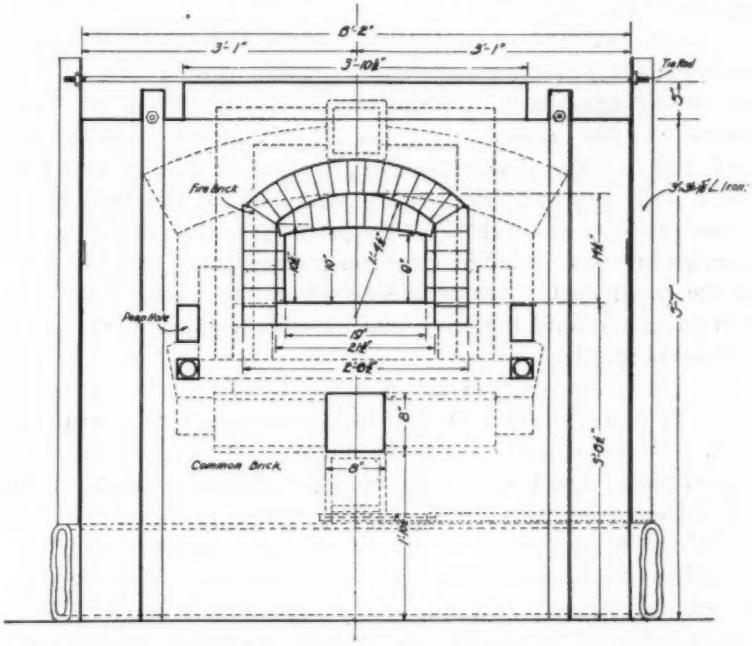
Section E-E

time these two dampers were opened wider to increase the cooling rate, until finally they were wide open. The furnace was then allowed to cool with no attention until the temperature was low enough to permit the door to be opened without the chance of dangerous temporary strain being set up in the glass.

The equipment used for temperature measurement consisted of Chromel-Alumel thermocouples, connected by compensating lead wire to a cold junction buried in the ground, and thence to a portable potentiometer. Actual measurements throughout the furnace proved that the temperature was so uniform that a single thermocouple encased in a one-half inch pipe extending through the top flue and crown into the furnace was sufficient. The end of the pipe was always lowered until it touched the glass. Tests

were made on the calibration of the couples at frequent intervals, although little variation was found and no trouble experienced from this source.

The results obtained by the use of this furnace have been quite satisfactory. An average furnace man, after a few days' experience, if provided with proper annealing schedules, could anneal one inch slabs to the grade of excellence called for in the regular commercial use of optical glass. By taking care in operating the



furnace, the author has succeeded in annealing large slabs two inches thick to about the practical limit of freedom from strain. A recent test of a slab annealed in this furnace showed a retardation of less than $2.5\mu\mu$ per cm when the glass was examined in the usual manner in polarized light between crossed nicols.

The author does not claim that the above design could not be improved upon. In fact several changes could be made that might result in fuel economy and greater ease of control. The insulation, for instance, is important enough to justify a 9″ wall

of insulating brick in place of the $4^1/_2''$ wall. The expense of this extra wall thickness might be lessened by using a $4^1/_2''$ insulating brick wall, together with an air space between the former and the common-brick shell. One would expect about as good results from the air space insulation as from the extra $4^1/_2''$ of insulating brick. At low temperatures, *i. e.*, below 200°C, an air space would be a very efficient insulator, and the exterior surface of the original $4^1/_2''$ Sil-O-Cel brick wall should be at a lower temperature than this.

To test this latter conclusion, temperature measurements were made on the wall temperature of a furnace of this type. With the annealing chamber held between 400° and 500°C, the temperature in the center of the Sil-O-Cel wall varied between 120° and 130°C. The temperature at the center of the common-brick wall averaged between 70° and 74°C at the same time. These readings were taken with a mercury-in-glass thermometer inserted in holes drilled in the proper distance so that the bulb of the thermometer was at the center of the brick. Lead foil was packed around the bulb, and asbestos packing filled in the remaining space.

Havard[1] states that an air space is a better insulator than a two-inch common brick wall, if the temperature of the hotter surface is below 625°C. This is quite evidently an error, as his figures were taken from a bulletin of the Bureau of Mines,[2] and the original gives this limiting temperature as 625°C *absolute*, or 565°F, ordinary scale. This latter is also confusing, as it was doubtless meant for 665°F. It is evident that below 350°C, ordinary scale, an air space is as good an insulator as two inches of red brick. This limiting temperature would be lowered considerably, if an insulating material were employed in place of the red brick. It is unquestionable, however, that the air space would be as efficient an insulator under the conditions mentioned above, as the same thickness of insulating brick.

Another alteration in the construction, which might increase the efficiency of the furnace, would be the use of 13″ tile in place

[1] Havard, "Refractories and Furnaces," McGraw-Hill Book Co., 1912.

[2] *Bulletin*, No. **8**, Bureau of Mines. "The Flow of Heat through Furnace Walls."

of 8″ in the flue, which takes the hot products of combustion from the chamber under the furnace floor. This flue could be made to run the entire length of the furnace before opening into the main flue leading to the stack. The main flue then must be placed at the front of the furnace. The 8″ auxiliary air flue, which has its inlet at the front of the furnace, could then pass through the inside of the 13″ flue tile, in such a manner that the heat in the latter would entirely surround the tile carrying the incoming cold air. The slight recuperative effect thus obtained ought to aid in maintaining a definite temperature and would undoubtedly result in economy in fuel.

Proper annealing schedules may be quite easily worked out from the information given by Adams and Williamson[1] if the annealing temperature of the glass is known. By using their method with this type of furnace, prisms and large blocks up to 10 cms in thickness have been satisfactorily annealed.

SPENCER LENS CO.
OPTICAL GLASS PLANT
HAMBURG, N. Y.

[1] L. H. Adams and E. D. Williamson, *J. Opt. Soc. Am.*, **4**, 213 (1920).

THE TRANSVERSE STRENGTH OF FIRE-CLAY TILES AT FURNACE TEMPERATURES[1]

By R. F. Geller

ABSTRACT

Commercial fire-clay tiles, were tested at furnace temperatures, for transverse strength. This was supplemented by tests of tiles of known composition made in the laboratory. Moduli of rupture obtained varied from 245.5 pounds per square inch at 1275°C, to 28.9 pounds per square inch at 1350°C but the data collected do not warrant definite conclusions.

Introduction

There has been in the past an insistent demand for data regarding the transverse strength of fire-clay tiles at furnace temperatures for the purpose of guiding engineers in the design of metallurgical and other furnaces. The present work was intended to supply information on this subject as far as possible.

The investigation consisted essentially in the testing of commercial fire-clay tiles of different makes for transverse strength at different temperatures supplemented by tests of tiles of definite composition made in the laboratory.

Experimental

Tiles were secured from four manufacturing concerns, situated in Colorado, Missouri, Kentucky and Pennsylvania, and were marked, respectively: C, M, K and P. Two standard sized tiles were obtained, one being 12x12x2 inches and the other 26x9x3 inches.

The tile C showed very little iron discoloration. The grog varied from approximately eight mesh to the finest grades. The structure was fairly dense and had every appearance of a well balanced proportion between calcined material and the bond clay. The refractoriness of this tile, as well as that of the others, was satisfactory the softening points being equivalent to cone 31 or above.

Tile M possessed a dense structure but was spotted throughout with iron stain and slag. Blisters of $^1/_8$ inch or more in diameter

[1] Received April 30, 1921. Published by permission of the Director, U. S. Bureau of Standards.

were of common occurrence and quite uniformly distributed, indicating large amounts of ferruginous impurities. The average color was a dark buff and the fracture seemed to indicate that the body was composed mainly of fine quartz and bond clay, although some grog as coarse as four mesh and over was present.

Tile K was made of a buff burning clay, the body containing a large amount of coarse material. It showed innumerable fine cracks of about $^1/_4$ inch in length, running parallel to the face of the tile and indicating a lack of fine grog, causing the bond to shrink away from the coarser particles.

The specimens marked P were composed of a white burning body showing no iron discolorations. Very little grog as coarse as eight mesh was in evidence and the structure as a whole was homogeneous and dense.

The method of testing followed in this work is similar to that given by the American Society for Testing Materials in its specifications for the determination of the resistance of fire-brick to load. Unless otherwise stated, the heating schedule given for heavy duty refractories in which the temperature is raised from 100°C to 1350°C in four and one-half hours was followed. The furnace used was of special construction and is shown in figure 1. It was first intended to fire two tiles simultaneously and a double loading device was provided. Owing to the uneven heating which the test specimens received, the furnace was altered so that only one tile could be tested. A baffle wall with checker work protected the tile from the direct flame and after a few trial runs the openings were so adjusted that the top and bottom sides of the specimen were practically uniform in temperature. Cones placed in various parts of the chamber showed no variation and these results were checked by means of a thermocouple. The special fire clay pieces used in supporting the tile and applying the load were made in the laboratory and consisted of

No. 1 fire-brick ground to pass the 10 mesh sieve	55%
Kentucky ball clay No. 4	15
Florida kaolin	15
Georgia kaolin	15

These were burned to cone 12 and gave very satisfactory service.

The furnace was covered with flat tiles 10x4 inches in cross section which at first caused considerable trouble by warping and cracking. This was due no doubt to the temperature gradient

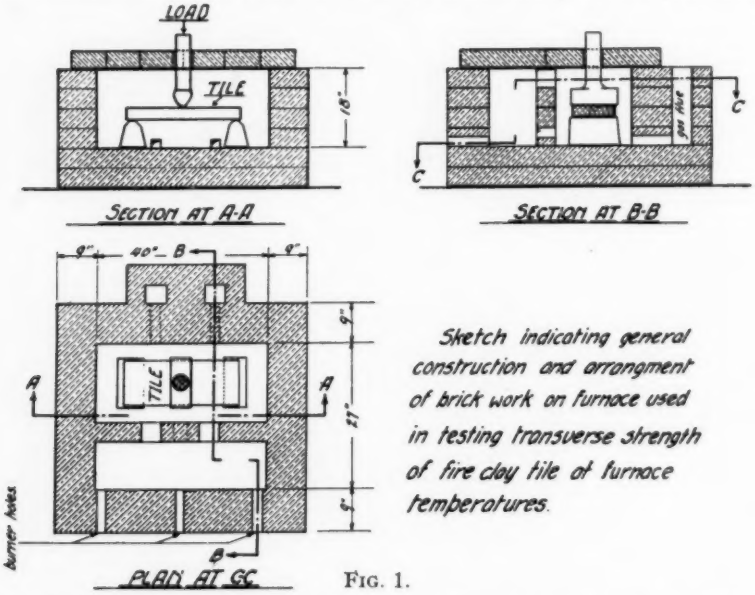


FIG. 1.

to which they were subjected, varying from 1350°C on the inside to approximately 100°C on the outside. Tiles of the following composition, burned to cone 12, were then made and no further trouble encountered:

Grog	(75% ground No. 1 fire-brick, 4 to 60 mesh)	60%
	(25% ground No. 1 fire brick thru 60 mesh)	
Tennessee ball clay No. 5		15
Georgia kaolin		15
Florida kaolin		10

The results obtained on specimens C are shown in table I.

In the first two burns the load was applied, until failure occurred, as soon as 1350°C was reached. The moduli of rupture lie fairly closely together, and when the load was applied at a lower temperature, as shown in the third burn, higher results were obtained, as would be expected. A greatly increased span (see runs 4 and 5)

TABLE I

Span in inches	Cross section, inches	Highest temp., °C	Load in pounds	Length of burn Hr.	Min.	Remarks
11	12″x2″	1350	84	4	30	M = 28.9 lbs./sq. in.
16	9″x3″	1350	113	4	37	M = 33.5 " " "
14	9″x3″	1325	209	4	15	M = 54.2 " " "
24	9″x3″	1325	20	4	15	Failed at this temp.
24	8¾″x3″	1275	20	4	00	" " " "
12	6″x2″	1320	20	4	20	" " " "
14	9″x3″	1300	20	6	30	Medium schedule Preheated to 425°C
6	6″x2″	1300	20	7	00	Medium schedule Preheated to 500°C

caused failure before the desired temperature was reached and the same was true in experiment 6 where the cross section was half of that in run No. 1.

It was then decided to preheat the tile slowly, to insure uniform thermal conditions, and to double the time element in the heating curve in order to guard against any strains resulting from a too rapid rise of temperature. As shown in runs 7 and 8, the tile actually proved weaker under these conditions. This would seem to indicate that failure occurred, not because of strains due to thermal changes, but to actual weakness of structure in the tile at high temperature. The load of twenty pounds was applied by means of the special fire-clay block which had this weight and which was used in breaking the tile. It was intended in the first place to serve for the transmission of the pressure from above. This weight rested on the specimen from the beginning of the burn.

No positive results were obtained from the tests of the tiles M. The data relating to this work are shown in table II:

TABLE II—TILE "M"

Span in inches	Cross section, inches	Highest temp., °C	Load in pounds	Length of burn Hr.	Min.	Remarks
12	11¾″x2″	1350	20	4	37	Failed at this temp.
20	9″x3″	1300	20	4	15	" " " "
12	9″x3″	1300	21	8	00	Medium schedule. Preheated 450°C
14	9″x3″	1275	20	7	30	Medium schedule. Preheated 450°C
12	11¾″x2″	1275	640	3	45	M = 245.5 lbs./sq. in.
24	9″x3″	1175	21	3	30	Failed at this temp.

The first two runs and the last resulted in failure. The first had not failed when 1350°C was reached although it had bent a little, but after holding seven minutes at this temperature, it gave way completely. With preheating and a more moderate firing schedule, shown in tests 3 and 4, the specimens had less strength, similar to tile C. In run No. 5, it was decided to apply a breaking load at a temperature of 1275°C since 1350°C appeared to be a critical point as related to the strength of the tile. A high modulus of rupture of 245.5 pounds per square inch was obtained, and is evidently due to the lower temperature employed.

Test pieces K composed of the buff burning body, containing a large proportion of coarse grog, gave rather poor results, as shown in table III:

TABLE III—TILE "K"

Span in inches	Cross section, inches	Highest temp., °C	Load in pounds	Length of burn Hr.	Min.	Remarks
16	9″x3″	1300	20	4	30	Failed at this temp.
18	9″x3″	1275	20	3	17	Failed at this temp. Preheated 275°C
14	9″x3″	1275	20	3	15	Failed at this temp. Preheated 275°C
24	9″x3″	1150	20	3	18	Slowly gave way at this temp.
$8^{2}/_{3}$	9″x3″	1350	447	5	30	Held at 1350°C for 1 hour. One-third of tile at each end held rigid in brick wall. M = 35.9 lbs./sq. in.

The first four burns resulted in failure before the maximum temperature of the heating curve was reached. As the data indicate, preheating again increased the weakness of the tile. It was then decided to alter the conditions of the test in order to more nearly approximate actual conditions under which tile are used. Accordingly, one-third of the tile at each end was firmly embedded in a brick wall. This left one-third of the tile, or eight and two-thirds inches, as a span between the walls. The temperature was raised to 1350°C as before, but held there for one hour before applying the critical load. A modulus of rupture of 35.9 pounds per square inch was obtained. The method of

testing described above was again applied to the tile P. In this case the specimen developed a modulus of rupture of 28.75 pounds per square inch which is less than that of tile K, although as the data given in table IV show, tile P in every other case appears to be stronger. Since the large mass of brick required to build the walls in this test caused an uneven distribution of heat, the results can not be considered of much significance and no more tests of this kind were made.

TABLE IV—TILE "P"

Span in inches	Cross section, inches	Highest temp., °C	Load in pounds	Length of burn Hr.	Min.	Remarks
18	9"x3"	1350	20	4	30	Failed
16	9"x3"	1350	464	5	00	M = 137.4 lbs./sq. in.
10	12"x2"	1350	83	5	00	M = 26.0 lbs./sq. in.
20	9"x3"	1325	20	4	15	Failed at this temp.
12	9"x3"	1300	20	8	00	Preheated to 450°C
10	6"x2"	1300	158	4	15	M = 98.7 lbs./sq. in.
6	6"x2"	1300	20	7	15	Preheated to 500°C. Failed
14	9"x3"	1175	20	7	00	Preheated to 275°C. Failed
8 2/3	9"x3"	1350	314.3	5	30	Held at 1350°C for 1 hour. 1/3 of tile at each end held rigid in brick wall. M = 28.75 lbs./sq. in.

Considering the other tests made on tile P, we again find, as shown in runs 5, 7 and 8, that preheating tends to weaken the tiles at the higher temperatures. Run No. 2 shows an abnormally high strength which can not be explained from the data at hand. The modulus of rupture obtained in test No. 3, however, agrees fairly well with that of tests 1 and 2, as shown in table 1. In run No. 6, the modulus of rupture of 98.7 pounds per square inch may not be inconsistent with others obtained considering that the tile was broken at 1300°C.

In order to test a specimen of known composition, a few tile were made at the Bureau of Standards. These tile burned to a very pale buff at cone 12 and were quite dense, the structure resembling that of tile C. They consisted of 40 per cent of silicious Arkansas fire-clay and 60 per cent grog. The grog was a calcined mixture of equal parts Kentucky ball clay No. 4 and Georgia

kaolin, ground to such fineness that 75 per cent graded from 10 to 60 mesh and 25 per cent passed through the 60 mesh screen. The data obtained relatively to these tiles are shown in table V.

TABLE V

Span in inches	Cross section, inches	Highest temp., °C	Load in pounds	Length of burn Hr.	Min.	Remarks
10	$2^{7}/_{8}''$x$8^{1}/_{2}''$	1350	20	8	00	Preheated 300°C. Held at 1350°C for 20 min.
$15^{1}/_{2}$	$2^{7}/_{8}''$x$8^{1}/_{2}''$	1350	75	7	30	Held at 1350°C for 1 hour. Load applied over $4^{1}/_{2}$ inch bearing surface
13	$2^{7}/_{8}''$x$8^{1}/_{2}''$	1350	150	8	00	Preheated 300°C. Load applied as above

Although a short span was used, the specimen in run No. 1 compares favorably with the commercial tile C considering that it was preheated and also held at 1350°C for twenty minutes before failure occurred. Tests 2 and 3 on the Bureau tile were made under somewhat different conditions. In place of knife edges, the tile were supported on bricks and the load also applied with a brick as the bearing surface. As the results show, considerably more strength was developed but it was obvious from the appearance of the fracture that the force exerted was not one of pure bending, but a combination of bending and shear.

In each burn the deflection of the tile at the center was observed by means of a cathetometer. A gradual lowering could be measured during the ten to fifteen minutes preceding the collapse of the specimen. After removing from the furnace the test pieces, however, showed no signs of softening or bending, failure in every case being due to a sharply defined break.

Conclusions

Sufficient data have not been obtained to warrant definite conclusions, but it would seem that:

(a) At the temperature of 1350°C commercial brick-clay tiles as now manufactured have very little transverse strength.

(b) Tiles containing comparatively fine grog, graded to produce a body having a minimum of voids, possess the greatest transverse strength at high temperatures.

(c) Transverse strength decreases rapidly with increase in temperature and vice versa as shown by the moduli of rupture obtained:

°C	Lbs. /sq. in.
1350	28.9
1350	33.5
1350	26.0
1325	54.2
1300	98.7
1275	245.5

(d) The test as now conducted is too rigorous to determine with sufficient accuracy the effect of small variations in the structure of the tile or the physical conditions of the test, *i. e.*, span, cross section and temperature.

The writer wishes to thank the manufacturers concerned for their interest in furnishing the tiles.

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In the preparation of abstracts, authors should be guided by the following rules, which are illustrated by the abstracts in THIS JOURNAL for February and March, 1921.* The **new information** contained in an article should first be determined by a careful analysis; then the subtitles should be formulated; and finally the text should be written and checked.

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1. **Material not new** need not be analyzed or described in detail; a valuable summary of a previous work, however, should be noted with a statement indicating its nature and scope.

2. **The subtitles should together include all the new information;** that is every measurement, observation, method, improvement, suggestion and theory which is presented by the author as new and of value in itself.

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4. **The text should summarize the authors' conclusions and should transcribe numerical results of general interest,** including those that might be looked for in a table of physical and chemical constants, with an indication of the accuracy of each. It should give all the information that anyone, not a specialist in the particular field involved, might care to have in his note book.

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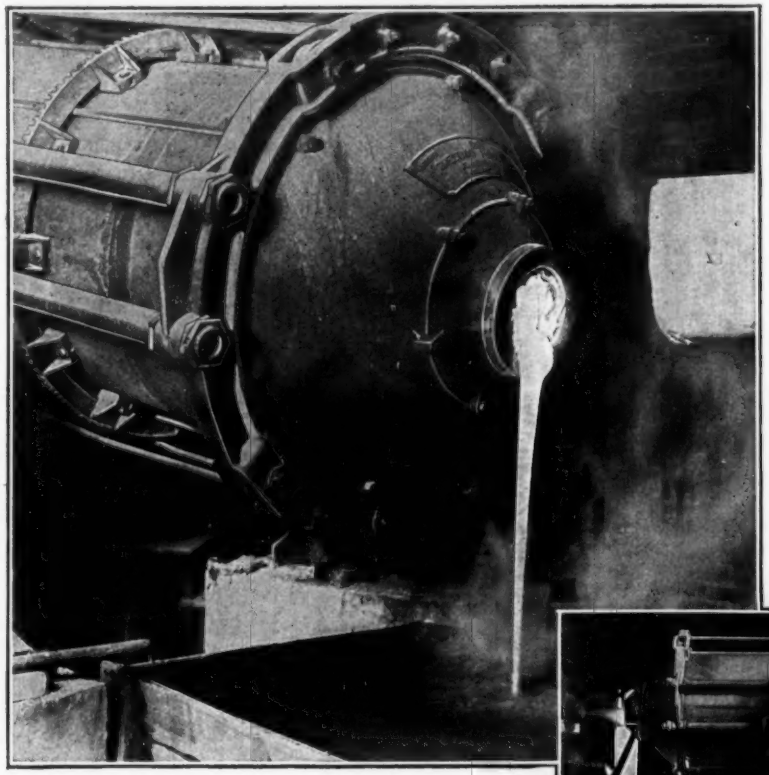
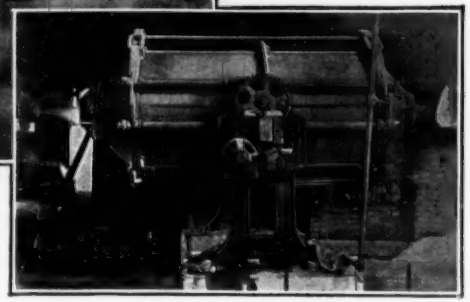
6. **Complete sentences** should be used except in the case of subtitles. The abstract should be made as readable as the necessary brevity will permit.

7. **The ms. of all abstracts must be typewritten and double or triple spaced.**

* The rules were prepared by the Research Information Service of the National Research Council. The Society is indebted to Dr. G. S. Fulcher of the Corning Glass Works (formerly with the National Research Council) for the rules and the illustrative abstracts.





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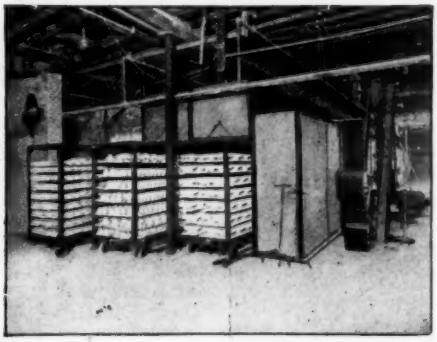

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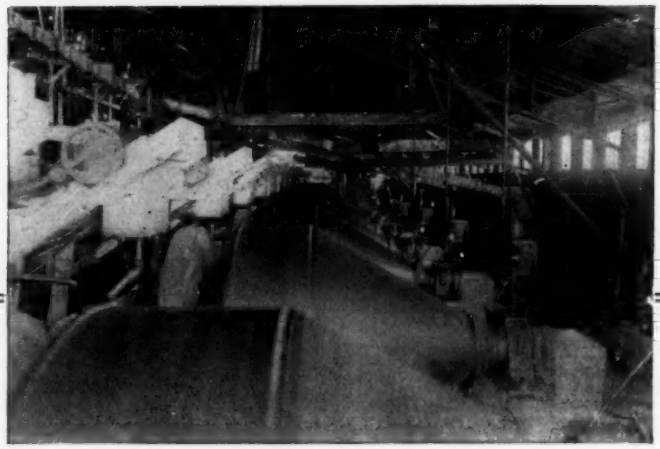

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	Page
(1) Abbé Engr'g Co., Outside back cover	
(2) Brown Instrument Co.	8
(3) Buckman and Pritchard, Inc.	13
(4) Celite Products Co.	14
(5) Central Maine Power Co.	12
(6) Chambers Brothers Co.	17
(7) Crossley Machine Co.	14
(8) Drakenfeld and Co., B. F.	10
(9) Edgar Brothers Co.	7
(10) Engelhard, Charles	11
(11) Exposition of Chemical Industries (Seventh)	18
(12) General Ceramics Co.	15
(13) Goebel & Co., J.	8
(14) Grand View Fire Clay Mines	7
(15) Hadfield-Penfield Steel Co.	7
(16) Hardinge Co.	4
(17) Harshaw, Fuller and Goodwin Co., Inside back cover	
(18) Manufacturers Equipment Co.	6
(19) Montgomery Porcelain Products Co	17
(20) Musiol, Charles	16
(21) O'Brien and Fowler	7
(22) Paper Makers Chemical Co.	9
(23) Pennsylvania Salt Mfg. Co.	17
(24) Philadelphia Drying Mch'y Co.	2
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(29) Surface Combustion Co.	12
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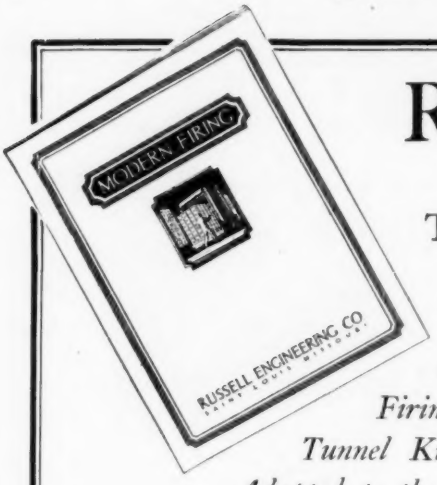

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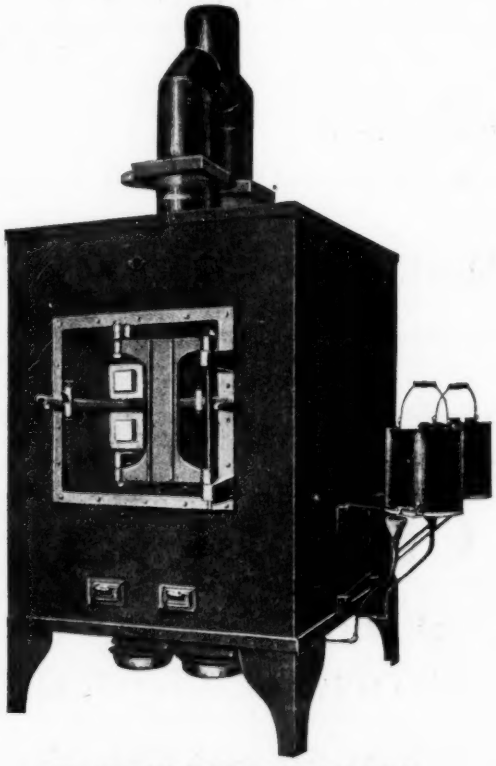

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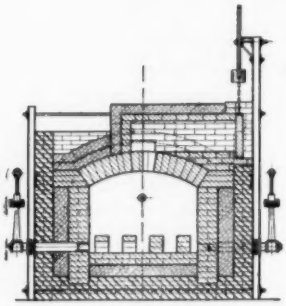

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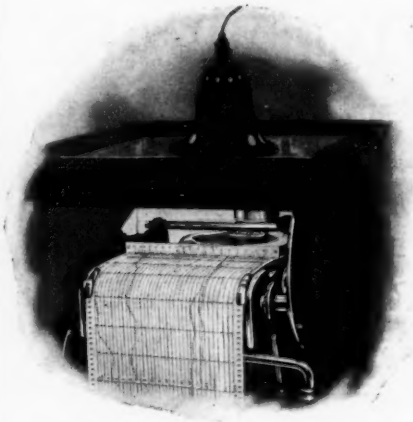

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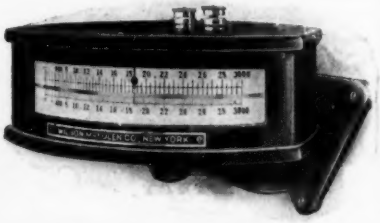

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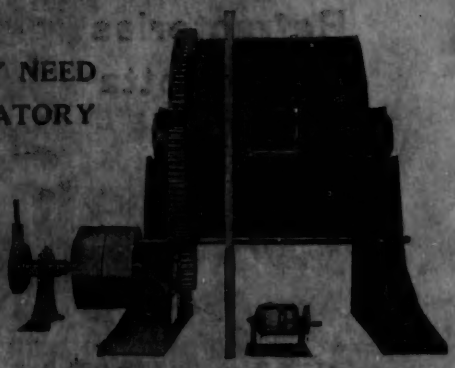

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